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The Journal *of the* Society of Dyers and Colourists

Volume 75 Number 4

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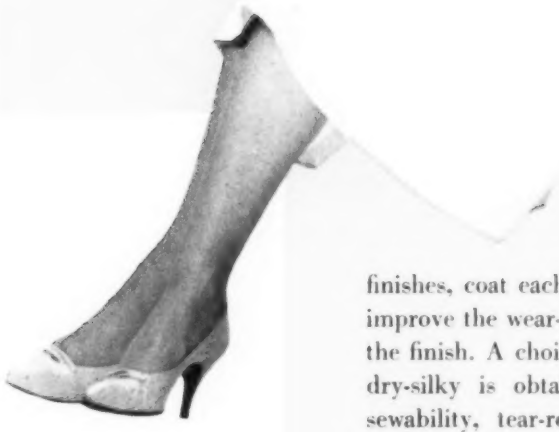

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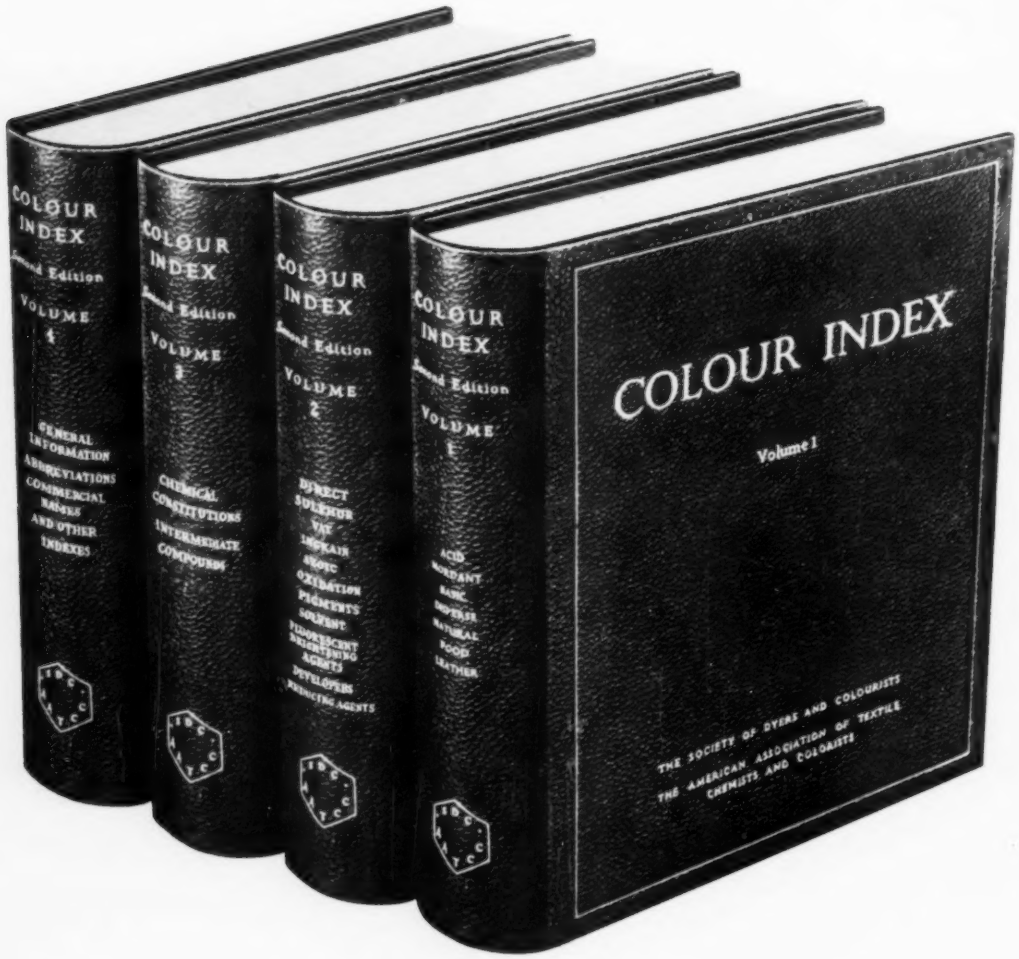

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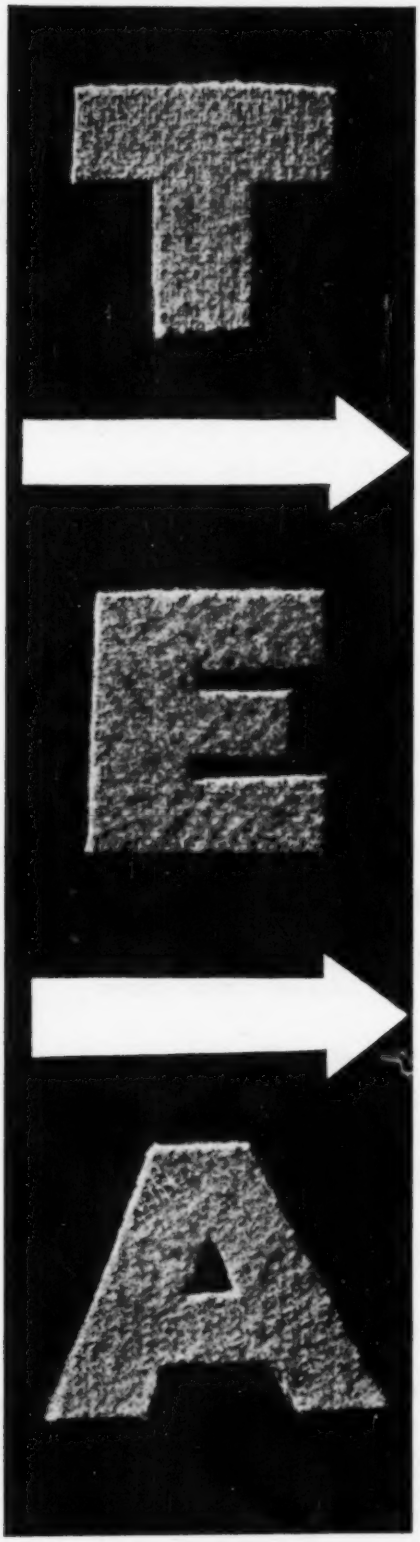

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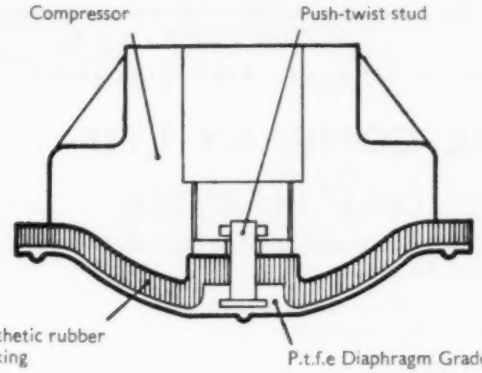

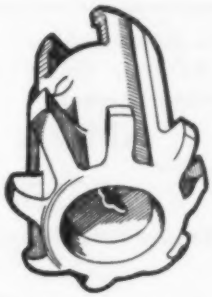

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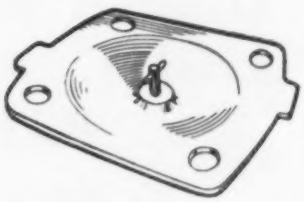

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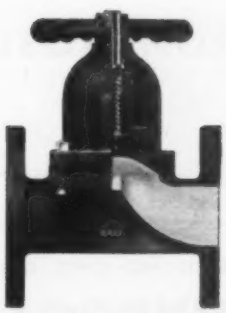

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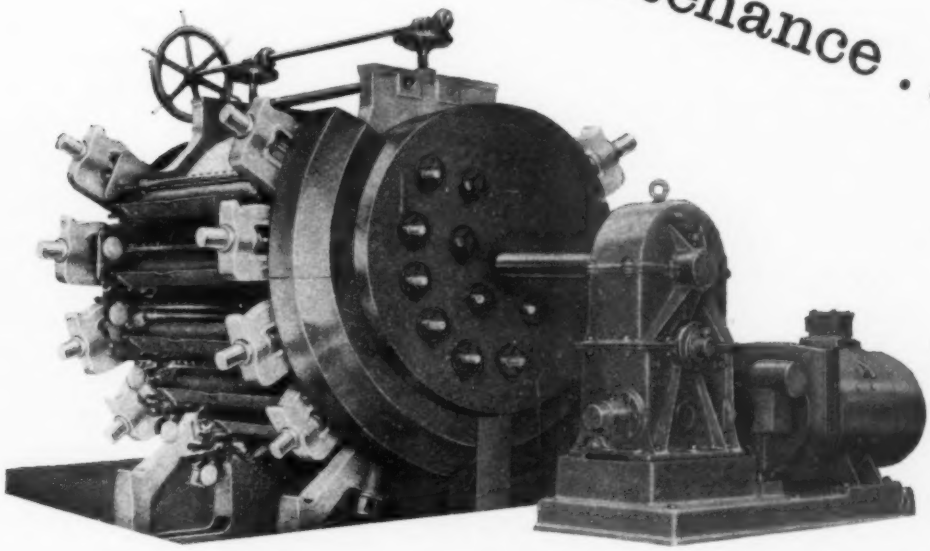

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Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1–10 of the January 1959 and pages 509–516 of the July 1958 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The Mechanism of the Coupling Process	*W. Bradley*
The Life and Times of Peter Griess	*W. H. Cliffe*
Felisol— An International Informative Label for Colour Fastness	*K. McLaren*
The Dyeing of Polyester Fibres and Blends	*H. W. Partridge*
The Mechanisms of Diazotisation	*J. H. Ridd*
Relative Rates of Reduction of a Series of Azo Compounds	*G. P. Warwick*

COMMUNICATIONS

Cellulose and Formaldehyde	*J. T. Marsh*
Setting Reduced Keratin Fibres with 1-Fluoro-2,4-dinitrobenzene	*P. T. Speakman*

EXPLANATORY PAPER ON MODERN THEORY

Surface Activity, Solution, and Adsorption	*C. H. Giles*

FORTHCOMING MEETINGS OF THE SOCIETY

Friday, 24th April 1959

LONDON SECTION. Annual General Meeting. Commander Dean Farnworth (United States Navy) will show and discuss his film *How the World Looks to a Colour-defective*. Waldorf Hotel, **London W.C.2.** 6 p.m.

Friday, 29th May 1959

MANCHESTER SECTION. *The Degradation of Chemically Modified Celluloses by Alkali*. Dr. W. M. Corbett (British Rayon Research Association). The Textile Institute, **Manchester.** 7 p.m.

Friday, 23rd October 1959

LONDON SECTION. Seventh London Lecture. Waldorf Hotel, **London W.C.2.** 7 p.m.

Friday, 12th February 1960

LONDON SECTION. Annual Dinner and Dance. Waldorf Hotel, **London W.C.2.** 7 for 7.30 p.m.

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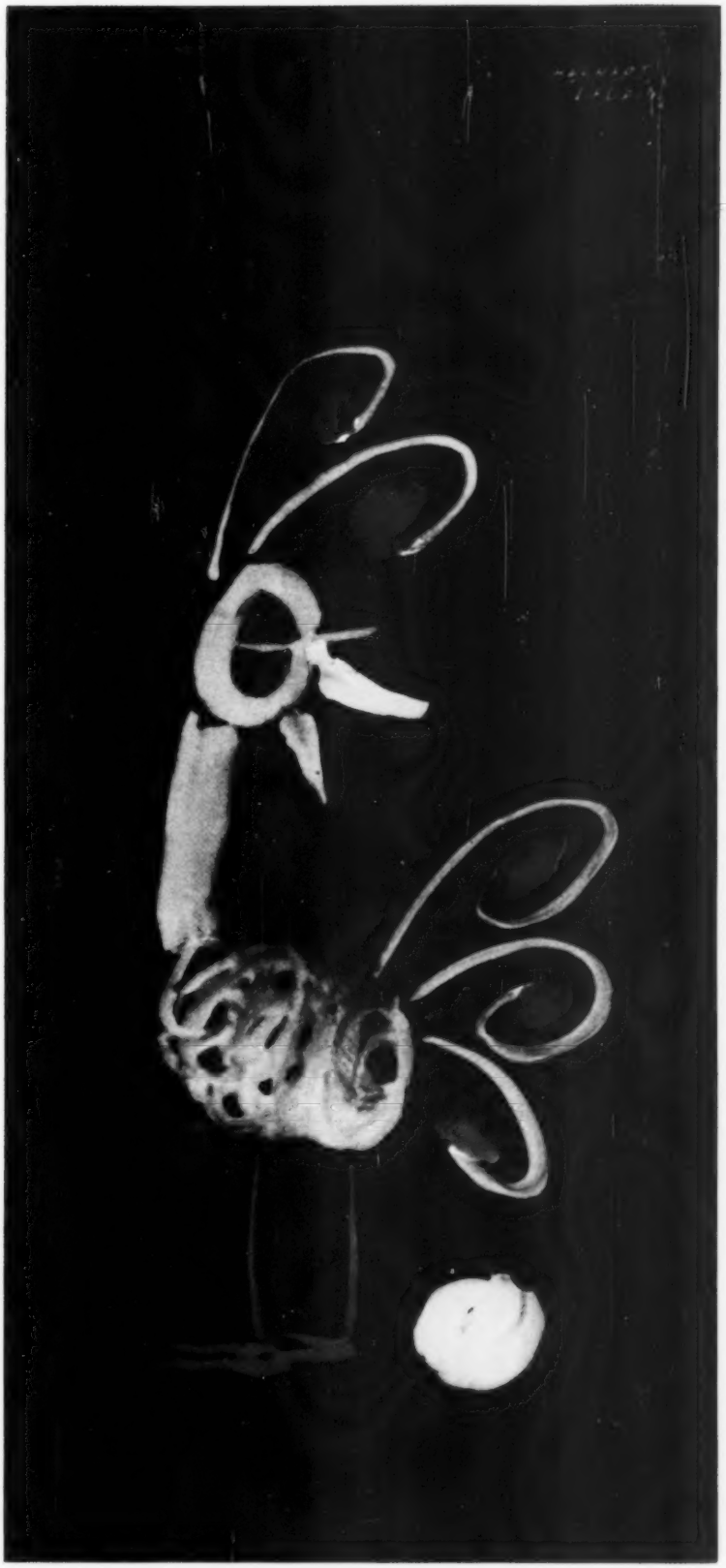

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Volume 75 Number 4 APRIL 1959 *Issued Monthly*

Proceedings of the Society

Acrylonitrile in the Textile Industry

J. H. MacGregor

Meeting of the Manchester Section held in the Textile Institute, Manchester, on 21st February 1958, Mr. J. W. Reidy in the chair

The rapid development of acrylonitrile as an industrial organic chemical during the past twenty years is reviewed and noted. Its reactivity with materials, such as cellulose, starch, wool, etc., which play a dominant rôle in the textile industry is discussed. The properties of the products obtained in these cyanoethylation reactions are considered, and some possible industrial applications of the products suggested. Finally a number of worthwhile uses of polyacrylonitrile and of copolymers containing acrylonitrile and other vinyl monomers are noted, with special reference to their outstanding value in the production of the comparatively new group of fully synthetic fibres known collectively as the acrylics. The properties of these fibres are commented on, and their value in certain applications is postulated. Brief mention is made also of the processing and dyeing of yarns and fabrics made from these fibres.

1. INTRODUCTION

Although the industrial chemistry of acrylonitrile began only some twenty years ago, it is almost sixty-five years since this compound was first prepared by Moureu[1]. By applying Dumas's classical method of preparing organic nitriles by dehydration of the corresponding amide, Moureu, in 1893, succeeded in preparing acrylonitrile by heating acrylamide with phosphorus pentoxide—

$$CH_2{:}CH{\cdot}CO{\cdot}NH_2 \xrightarrow{P_2O_5} CH_2{:}CH{\cdot}CN + H_2O$$

For forty years after its discovery acrylonitrile remained in the shadows, and very little interest was shown in its chemistry. Indeed, it was not until about the middle of the 1930s that the intensive search, mainly in Germany, for synthetic rubberlike substances led to the consideration of acrylonitrile.

It had been shown about that time that butadiene $CH_2{:}CH{\cdot}CH{:}CH_2$ could be polymerised by emulsion methods to give products which were rubberlike in appearance. These products, however, fell far short of natural rubber in almost every other respect. Nevertheless, it was soon discovered that, when copolymerised with certain vinyl derivatives such as acrylonitrile or styrene, butadiene gave very promising rubberlike materials. Indeed, a butadiene–acrylonitrile copolymer (4:1 by weight) possesses oustanding resistance to hydrocarbon solvents as well as to oxidation and to heat.

When the supply of natural rubber became very restricted during the Second World War it was not surprising that immense quantities of synthetic rubber, based on butadiene, were required by the armed forces of the various combatants. In view of the very desirable properties of the butadiene–acrylonitrile synthetic rubber its large-scale production was given high priority. In the United States, with its immense resources of natural gas, huge plants were erected to produce the butadiene–acrylonitrile copolymer. Consequently, acrylonitrile was produced on a substantial scale. In Germany, also, the need for the copolymer led to the large-scale manufacture of acrylonitrile.

This striking initial development in the industrial history of acrylonitrile has led naturally to its availability in large quantities. Today, it is by far the most important individual nitrile and is undoubtedly one of the most versatile organic chemicals which have become available commercially within the past quarter of a century.

Besides its importance in the high-polymer field, where it is not only a component of one of the most useful synthetic rubbers but also the main constituent of a wide and ever growing range of commercially available synthetic fibres, known collectively as the *acrylics*, acrylonitrile is also capable of interacting with a large number of organic substances containing reactive hydrogen atoms.

Before describing and discussing these recent developments of acrylonitrile insofar as they affect the textile industry, it may be of interest to consider very briefly the two main methods by which it is prepared commercially.

2. MANUFACTURE OF ACRYLONITRILE

There are two main synthetic routes to the large-scale production of acrylonitrile. The first is based

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on the dehydration of ethylene cyanohydrin, which is itself conveniently prepared by the addition of hydrogen cyanide to ethylene oxide in the presence of a basic catalyst such as diethylamine. Dehydration of ethylene cyanohydrin is accomplished smoothly and in good yields (80–90%) either in the vapour phase, by passing it over active alumina at 300°C., or in the liquid phase at the boil under atmospheric pressure, using such diverse catalytic substances as aluminium powder, metallic tin, magnesium carbonate, or sulphamic acid—

$$\underset{\diagdown O \diagup}{CH_2{-}CH_2} \xrightarrow{+\ HCN} \underset{CH_2{\cdot}CN}{\overset{|}{CH_2OH}} \xrightarrow{-\ H_2O} \underset{CH{\cdot}CN}{\overset{\|}{CH_2}}$$

The second method of manufacture, which is claimed to be the cheaper, entails the addition of hydrogen cyanide to acetylene. Vapour-phase reaction at 400–500°C. over barium peroxide catalyst is uneconomic for several reasons, but mainly because yields are low. The liquid-phase, atmospheric-pressure, low-temperature reaction, using a Nieuwland-type catalyst (ammonium chloride–cuprous chloride solution), requires a large excess of acetylene (10 moles of acetylene per mole of hydrogen cyanide). The reaction is exothermic, and sufficient heat is liberated to maintain the reaction mixture at 80°C., at which temperature the highest yields are obtained. Yields of pure acrylonitrile of 80% based on acetylene and of 85% based on hydrogen cyanide are reported to be obtainable by this process—

$$CH{:}CH + HCN \longrightarrow CH_2{:}CH{\cdot}CN$$

Acrylonitrile is a colourless liquid which boils at 77–78°C. It has a low flash point and its vapours form explosive mixtures with air. It is toxic by inhalation, by absorption through the skin, and by oral ingestion. Great care has, therefore, to be exercised in handling acrylonitrile.

3. CHEMICAL BEHAVIOUR OF ACRYLONITRILE

It is well known that the double bond in olefins is unreactive to anionoid reagents because it cannot act as an electron acceptor. When a substituent is present, however, that influences the electronic configuration of the molecule by conjugation, the C:C bond becomes sensitised to anionoid additions.

In acrylonitrile we have a typical example of such a condition. As the nitrile group is electron-attracting and conjugated with the double bond, electronic shifts occur tending to produce the mesomeric ion—

$$CH_2{=}CH{-}C{\equiv}N \longleftrightarrow \overset{+}{C}H_2{-}CH{=}C{=}\overset{-}{N}$$

This facilitates ready attack by an anionoid group X^- on the relatively positive carbon atom. The reaction is completed as outlined below—

$$X^- + \overset{+}{C}H_2{-}CH{=}C{=}\overset{-}{N} \longrightarrow X{-}CH_2{-}CH{=}C{=}\overset{-}{N}$$

$$X{\cdot}CH_2{\cdot}CH{=}C{=}\overset{-}{N} \xrightarrow{H{-}OH} X{\cdot}CH_2{\cdot}CH_2{\cdot}CN + OH^-$$

X^- is readily produced by the action of a strong base on compounds containing reactive hydrogen atoms—

$$R{\cdot}H + OH^- \longrightarrow R^- + H_2O$$

Thus all organic compounds containing reactive hydrogen atoms, e.g. alcohols, water, amines, react readily with acrylonitrile in the presence of a base. However, while alcohols will react only in the presence of a basic catalyst, some amines react with acrylonitrile in the absence of catalyst, or even under acid conditions.

Indeed, MacGregor and Pugh [2] have shown that in the presence of small amounts of strong alkaline condensing agents, e.g. sodium hydroxide, aliphatic alcohols add to acrylonitrile to give β-alkoxypropionitriles—

$$R{\cdot}OH + CH_2{:}CH{\cdot}CN \longrightarrow R{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}CN$$

The reaction, involving the introduction of the β-cyanoethyl ($-CH_2{\cdot}CH_2{\cdot}CN$) group into organic substances containing reactive hydrogen atoms, is known as *cyanoethylation*. Many well known substances of great importance and of widespread application in the textile industry, such as cellulose, starch, polyvinyl alcohol, and wool, contain many reactive hydrogen atoms in their giant molecules. It is now proposed to consider the interaction of acrylonitrile with these substances; all of the reactions discussed embody the introduction of β-cyanoethyl groups. The properties and potential applications of these cyanoethylated substances will also be reviewed and discussed.

3.1. Cellulose

Cellulose in all its chemical reactions behaves as if three hydroxy groups in every glucose residue were free: these groups, as is well known, react in ways typical of alcohols in the formation of triesters and triethers. Under appropriate conditions, part or all of the hydroxy groups in cellulose can be cyanoethylated by interaction with acrylonitrile.

It has been found [3] that cyanoethylation reactions with cellulose produce more uniform products and yield more reproducible results if the reactions are carried out when the cellulose is in solution or passes into solution early in the reactions. For this reason viscose, which contains about 8% of cellulose and 6% of sodium hydroxide, is usually employed as the source of cellulose.

When acrylonitrile is added to viscose there is a very rapid colour change, the original golden brown colour of the viscose changing within a very few minutes to an olive green. This colour change is caused by the interaction of the acrylonitrile with the elements of hydrogen sulphide in alkaline solution to form $\beta\beta'$-thiodipropionitrile (di-2-cyanoethyl sulphide)—

$$NC{\cdot}CH{:}CH_2 + H_2S + CH_2{:}CH{\cdot}CN \longrightarrow NC{\cdot}CH_2{\cdot}CH_2{\cdot}S{\cdot}CH_2{\cdot}CH_2{\cdot}CN$$

If the reaction is carried out at low temperatures (+ 16°C.) the dinitrile can be removed from the reaction mixture by filtering. Hydrolysis of the dinitrile yields $\beta\beta'$-thiodipropionic acid. This interesting dibasic acid and its derived sulphone can be condensed with diamines and glycols to

yield sulphur-containing polyamides and polyesters. The polyamides decompose at their melting point and appear to be unsuitable as fibre-forming polymers. The polyesters, on the other hand, are viscous oils. Esters of the acid, however, are valuable plasticisers for polyvinyl chloride and synthetic rubbers. These plasticisers are characterised by their excellent resistance to low temperatures.

3.1.1. Water-insoluble, Alkali-soluble Ethers—When the molar ratio of acrylonitrile to anhydroglucose in viscose is 0·5–1·2, ethers soluble in dilute aqueous solutions of sodium hydroxide and insoluble in water are produced. They contain 0·2–0·3 cyanoethyl group per anhydroglucose unit. These ethers can be spun into filament and staple fibre yarns in the same way as viscose. The yarns have a high water imbibition, ca. 300%, and swell strongly in water but do not dissolve. They dissolve in 1·25% sodium hydroxide solution, and consequently can be used as scaffolding threads for cellulosic yarns such as cotton. The yarns will also dissolve in 25% aqueous ammonium thiocyanate solution; this solution is, of course, necessary for dissolution of the yarns when they are used as scaffolding threads in conjunction with wool. Cyanoethylcellulose yarns of this type may also be used successfully as a substitute for the drawthread in sock manufacture.

Cyanoethylcellulose staple fibres may be carded with viscose rayon staple fibres or cotton fibres to form webs, and advantage taken of the solubility of the cyanoethylcellulose fibres in dilute aqueous solutions of sodium hydroxide to form non-woven bonded-fibre fabrics [4].

The ethers of this group may also be obtained in the form of irregular fibrous lumps by acidification of the viscose–acrylonitrile reaction mixture. After washing free from acid and then drying, the ethers, in this form, swell and dissolve readily in dilute aqueous solutions of sodium hydroxide. For example, solutions containing 5% of the ether and 3% of sodium hydroxide, and 8% of the ether and 5% of sodium hydroxide, can be quickly and easily prepared; they remain stable for long periods if kept at normal room temperatures. These solutions can be used for the fixation of pigments, for the production of wash-fast, linen-like finishes on fabrics woven from viscose staple fibre yarns, as thickeners for printing pastes, for the production of novelty print effects, and for the production of bonded-fibre fabrics.

3.1.2. Water-soluble Ethers—When the molar ratio of acrylonitrile to anhydroglucose in viscose is 1·5–4·0, water-soluble ethers are obtained.

Water-soluble ethers may also be prepared from chopped waste viscose rayon yarns by swelling them first by mixing with a solution containing urea and sodium hydroxide in a Pfleiderer or Beken type of mixer. Sufficient acrylonitrile is then added to the mixture to give the alkali-soluble ether on further mixing. When this is accomplished, a further amount of acrylonitrile to give a total of one mole of acrylonitrile per mole of anhydroglucose in the regenerated cellulose is added, and mixing continued until the water-soluble ether is formed (usually 2 hr.). After neutralisation of the reaction mixture the ether solution is purified by dialysis. This is perhaps the most economical method of preparing cyanoethylcellulose ethers of the water-soluble type. They are purified most satisfactorily by dialysis of the acidified reaction mixture and can be obtained in the dry state by precipitation with ethanol or acetone. The ethers are soluble in acidic, neutral, and alkaline aqueous solutions. Aqueous solutions deposit transparent, tough, pliable colourless films on drying. The films readily dissolve in water, but are extremely resistant to hydrocarbon solvents: they behave like films of polyvinyl alcohol in this respect. Water-insoluble films may be produced by drying solutions of the water-soluble cyanoethylcelluloses containing small amounts of urea–formaldehyde or melamine–formaldehyde precondensates, and the necessary amount of a suitable catalyst to assist cross-linking and resin formation.

The water-soluble cyanoethylcelluloses are very good emulsifying agents. Their aqueous solutions, when mixed with a suitable lubricant such as tallow, are excellent sizes for warps of viscose rayon staple yarns: in addition to providing very satisfactory protection to the warp yarns during weaving, they are also readily removed from the fabrics woven from them during subsequent scouring.

3.1.3. Acetone-soluble Ethers—As has been explained elsewhere [3], acetone-soluble cyanoethylcelluloses cannot be prepared by the interaction of acrylonitrile and viscose. The ethers of this class are, however, readily prepared by first soaking a partly cyanoethylated cellulose (of either water-insoluble or water-soluble type) or viscose rayon yarn with a cold 1–2% aqueous sodium hydroxide solution and removing the excess liquor. The treated material is then refluxed with excess acrylonitrile. These ethers dissolve in the excess of acrylonitrile as they are formed. They contain 2·5–3·0 cyanoethyl groups per anhydroglucose unit and are precipitated from the acrylonitrile solution, after neutralisation of the reaction mixture, by addition of ethanol. The acetone-soluble cyanoethyl ethers can be dry-spun into filament yarns which have a tenacity similar to that of secondary cellulose acetate yarns. An outstanding characteristic of the acetone-soluble cyanoethylated yarns is their excellent resistance to photodegradation, a property not usually exhibited by other types of cellulose ethers. Triscyanoethylcellulose has an unusually high dielectric constant (13·3 at 60 cycles/sec. and 25°c.), which is attributed to rotation of the cyanoethyl group [5].

Yarns of triscyanoethylcellulose possess a subdued lustre and a soft silk-like handle. These yarns have good affinity for disperse dyes, but the colours are generally flatter than on secondary acetate yarns. Basic dyes applied direct or as cationic dyes give solid bright colours. Direct cotton dyes have very poor affinity. Acetone-soluble cyanoethylcelluloses are also good internal plasticisers for secondary cellulose acetate yarns.

3.2. *Starch*

As starch also contains three hydroxyl groups per anhydroglucose unit, it is not surprising that it should interact with acrylonitrile in the presence of an alkaline catalyst to produce cyanoethyl ethers.

Unlike cellulose, however, water-soluble ethers are obtained initially when the molar ratio of acrylonitrile to the anhydroglucose unit in starch is 0·1–1·5, and alkali-soluble, water-insoluble ethers are not obtained until this ratio is > 2. This obtains, of course, when the starch is in solution. Acetone-soluble cyanoethyl starch ethers can be obtained only by refluxing partly cyanoethylated starches with excess acrylonitrile in the presence of a small amount of a strong alkaline catalyst such as sodium hydroxide.

The water-soluble cyanoethylstarches possess good emulsifying and dispersing properties. Like the corresponding cellulose ethers they are satisfactory sizing materials and are especially useful as sizes for warps of viscose rayon staple yarns.

The water-insoluble, alkali-soluble ethers have been used successfully for applying, by padding processes, pigments to textile fabrics. The tris-cyanoethylstarches are soluble in acetone and other solvents. On drying, these solutions deposit tough but rather sticky films which have good adhesive properties.

3.3. *Polyvinyl Alcohol*

Owing to the closely similar colloidal behaviour of polyvinyl alcohol and starch, the reaction between acrylonitrile and polyvinyl alcohol in the presence of strong alkaline condensing agents proceeds along similar lines to that between acrylonitrile and starch. The initial products of the reaction are water-soluble, but as the molar proportion of acrylonitrile increases, water-insoluble, alkali-soluble cyanoethyl ethers are obtained. The water-soluble cyanoethyl ethers of polyvinyl alcohol deposit tough, pliable, transparent films on drying their aqueous solutions. These films are extremely resistant to hydrocarbon solvents. Like the corresponding cellulose and starch ethers, the water-soluble cyanoethyl ethers of polyvinyl alcohol are useful sizing materials.

Cyanoethyl ethers of polyvinyl alcohol soluble in solvents such as acetone cannot be prepared from aqueous solutions of the polyvinyl alcohol. They are readily prepared, however, by refluxing polyvinyl alcohol or a partial cyanoethyl ether of the alcohol with excess acrylonitrile in the presence of a small amount of aqueous sodium hydroxide. These ethers dissolve as they are formed in the excess of acrylonitrile, and may be isolated by pouring the neutralised reaction mixture into aqueous ethanol. On drying acetone solutions of these triethers, plastic and rather sticky films possessing good adhesive properties are deposited.

3.4. *Heterogeneous Cyanoethylation*

So far, only the homogeneous cyanoethylation of cellulose and similarly constituted substances has been considered. Reference is now made to work done on the heterogeneous cyanoethylation of cellulose.

If viscose rayon yarns are soaked in 1% aqueous sodium hydroxide solution for 5 min., the excess liquor removed by squeezing, and the treated yarns are then placed in acrylonitrile–benzene (50:50) maintained at 20°c., cyanoethylation takes place over a period of hours.

Initially, the interaction of acrylonitrile and regenerated cellulose in a two-phase system proceeds by swelling the cellulose chains, as is shown by increased water imbibition and increased absorption of direct dyes by the yarns. A certain amount of cyanoethylation also occurs in the early stages, but this is dwarfed by the extent of swelling which occurs. Later, however, cyanoethylation plays a more dominant part, with the result that water imbibition and absorption of direct dyes decrease, with simultaneous increase in the absorption of acid dyes. As the reaction proceeds over long periods (say after 24 hr.), hydrolysis of cyanoethyl to carbamoylethyl and carboxyethyl groups takes place, with reduction in the absorption of acid dyes.

The cyanoethylation of cotton has been the object of intensive research during recent years in the U.S.A.[6]. Cotton, having a more crystalline structure than regenerated cellulose, is more difficult to cyanoethylate. Nevertheless, by first soaking it in dilute aqueous sodium hydroxide and then allowing the treated cotton to interact with acrylonitrile, a partially cyanoethylated cotton, which has the same physical appearance and morphological structure as the original cotton, is produced.

Both temperature and time play a very important part in the reaction. For example, on soaking the cotton in 2% aqueous sodium hydroxide and then treating it at 55°c. with acrylonitrile, the nitrogen content of the cotton reaches an equilibrium of about 3·5% after about 30 min.; on carrying out the reaction at the boiling point of acrylonitrile, this equilibrium is reached in 3 min.

Two main processes for cyanoethylating cotton appear to have been developed in the U.S.A. The first utilises standard package-dyeing machine equipment with, of course, modifications to permit the safe handling of the acrylonitrile. For every part of cotton in the kier a mixture of 0·83 part of 8% aqueous sodium hydroxide, 6·6 parts of acrylonitrile, and 0·1 part of a wetting agent (e.g. Aerosol OT— American Cyanamid Co.) is added and circulated at about 25°c. for 20 min. The temperature is then raised to 38°c. for 30 min. After acidification with phosphoric acid to stop the reaction, excess acrylonitrile is recovered by countercurrent washing and distillation.

The second process involves continuous padding of the cotton with 6% aqueous sodium hydroxide followed by exposure to acrylonitrile vapour in a reaction chamber at 72°c. for about 3½ min.

Production costs are stated to be about 13–16 cents per lb. of cotton treated by either method.

A serious problem in these processes and indeed in most cyanoethylation reactions is the loss of acrylonitrile in a side-reaction with water in which bis-2-cyanoethyl ether is formed—

$$CH_2{:}CH{\cdot}CN + H_2O \longrightarrow HO{\cdot}CH_2{\cdot}CH_2CN \quad (i)$$

$$CH_2{:}CH{\cdot}CN + HO{\cdot}CH_2{\cdot}CH_2{\cdot}CN \longrightarrow NC{\cdot}CH_2{\cdot}CH_2{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}CN \quad (ii)$$

Raising the concentration of sodium hydroxide to increase swelling, and hence the reactivity of the cotton cellulose, has the disadvantage of causing hydrolysis and cleavage of cyanoethyl groups with consequent losses in the microbiological resistance of the cyanoethylated cotton. Moreover, anionic polymerisation of acrylonitrile becomes an important factor and a serious possibility at alkali concentrations higher than about 15%. Finally, bis-2-cyanoethyl ether formation is also alkali-dependent.

A new and interesting approach to the problem of overcoming the loss of acrylonitrile in side-reactions during the cyanoethylation of cotton has recently been reported by Bikales *et al.*[7], who use concentrated solutions of salts having the ability to swell cotton cellulose. When these salts are used together with dilute alkali, striking increases in the rate and extent of cyanoethylation of cotton have been obtained. According to these authors, the most effective salts are sodium thiocyanate and sodium iodide. Neither of these salts interferes with the properties of the cyanoethylated cotton obtained.

Partially cyanoethylated cotton fibres, known commercially in America as *Azoton*, have a nitrogen content of about 3·8%. It is claimed that they are similar in appearance, handle, and processing behaviour to untreated cotton. Some of the outstanding and characteristic properties of these fibres are—

(*a*) Permanently rot- and mildew-proof

(*b*) Stability and resistance to degradation by wet and dry heat

(*c*) Stability and resistance to degradation by organic and mineral acids

(*d*) Lower moisture regain: typical data are 5·0–5·5% compared with 7·0–7·5% for cotton

(*e*) Modified dyeing properties. For example, they have a greater rate of absorption of most classes of dyes than cotton. They can be dyed satisfactorily with acid and disperse dyes

(*f*) Improved stress–strain fibre properties compared with cotton

(*g*) Improved electrical properties. It is claimed that the dielectric constant increases proportionally to the degree of cyanoethylation.

3.5. Wool

As wool contains amine, amide, and disulphide groups, it is to be expected that it will react readily with acrylonitrile. Early work showed that cyanoethylation took place more easily if the disulphide linkages were reduced to thiol groups.

Bikales *et al.*[8], working along these lines, have succeeded in introducing significant amounts of cyanoethyl groups into wool without affecting adversely the physical appearance and properties of the fibre. Essentially, the wool is soaked in dilute sodium hydroxide solution ($<$ 0·4%) at room temperature for about 10 min., the excess alkali removed, and the wet wool treated at 60°C. with acrylonitrile in a package dyeing machine for 1 hr. After being neutralised with dilute acetic acid and then washed free from acid, the treated wool contains about 3–4% by weight of cyanoethyl groups, as determined by the increase in nitrogen content of the wool. It is claimed that the wool is not degraded by the treatment.

Cyanoethylated wool is dyed readily with cationic or direct dyes (applied at pH 4·3 $\pm$ 0·4 using formic acid). It has been stated that cyanoethylated wool dyes more rapidly with these types of dyes than untreated wool, and that colours about three times as deep are obtained.

4. DEPOSITION OF POLYACRYLONITRILE IN RAYON AND WOOL

During recent years much attention has been directed by Speakman and his co-workers [9] to the formation of deposits of vinyl polymers inside textile fibres. Indeed, it has been shown that under suitable conditions acrylonitrile can be polymerised inside rayon and wool fibres. A satisfactory method of bringing about these depositions inside textile fibres is first to impregnate the fibre with a solution of ammonium ferrous sulphate and dry. The fibres are then treated with an aqueous solution of hydrogen peroxide and acrylonitrile, whereupon polymerisation of the acrylonitrile is initiated by the free hydroxyl radical liberated in the reaction—

$$Fe^{2+} + H_2O_2 = Fe^{3+} + OH^- + {\cdot}OH$$

The incorporation of polyacrylonitrile in filament viscose rayon yarns [10] increases their stiffness, and as more polymer is deposited the filaments become increasingly distorted. About 8% of polymer on the weight of yarn may be introduced, however, without causing any changes in the diameter of the fibres.

In the case of wool Valentine [11] has shown that internal deposits (20%) of polyacrylonitrile render the wool virtually shrink-resistant. In addition, resistance to abrasion is improved by about 20%, while resistance to solvents also is increased. Moreover, the elastic properties are unimpaired, and the moisture regain is lowered (11·5% at 65% R.H.).

5. WATER DISPERSIONS OF BUTADIENE–ACRYLONITRILE COPOLYMERS

Reference has already been made to the successful industrial applications of a butadiene–acrylonitrile copolymer as a synthetic rubber. Within recent years, however, latices of a group of butadiene–acrylonitrile copolymers have become available commercially and have found wide application in many industries.

In the textile industry their good adhesive and film-forming properties and their excellent resistance to hydrocarbon solvents and oils have led to their growing application either alone or in association with other materials with which they are compatible. As finishing materials for textile fabrics and as warp sizes for loom-state fabrics they would appear to offer considerable scope for further development.

For example, they have been recommended as additives to urea–formaldehyde and melàmine–formaldehyde precondensates in finishing processes. It is claimed that, when used in conjunction with either of these amino resins, they improve appreciably the resistance of the treated fabrics to tearing and abrasion without crease-resistance and dimensional stability being affected.

As sizing materials, they may be mixed with starch or modified starch solutions, and the mixtures used as sizes for warps of cotton and rayon staple yarns required for the production of loom-state fabrics which are especially required to possess good tear and abrasion resistance. Warps of these yarns sized with such mixtures weave well.

The butadiene–acrylonitrile dispersions when used in conjunction with a suitable lubricant and penetrant have been recommended as suitable sizes for warps of spun-dyed filament cellulose acetate yarns. The warps weave well, and loom-state fabrics possessing a pleasing handle and good spot-proof properties are obtained. The fabrics are also commercially fast to dry-cleaning solvents, especially those based on hydrocarbons.

6. POLYACRYLONITRILE AND ITS COPOLYMERS

While many of the reactions and products already referred to can only be described as having potential uses in the textile industry, it now seems appropriate to consider a field of the industry where acrylonitrile has made and is making substantial strides. This refers, of course, to its importance as a raw material for the production of acrylic fibres.

The availability of acrylonitrile at the end of the Second World War has already been mentioned, and has indeed been likened to the large stocks of cellulose acetate dope which were available at the end of the First World War. Whether or not any significance may be attached to this fact, there is no doubt that in each instance a new fibre was produced and developed.

Acrylonitrile is readily polymerised. Indeed, polyacrylonitrile is probably the most singular of the whole series of vinyl polymers. No matter whether acrylonitrile is polymerised by block, solution, or emulsion methods, with the aid of heat, peroxides, or other radical-formers, it is almost invariably precipitated as a white amorphous crumbly mass, which can be easily pulverised to a white powder. It is not thermoplastic and has no definite melting point: on heating it becomes yellow at about 300°C., and decomposes at approx. 350°C. Unlike other vinyl polymers, polyacrylonitrile is insoluble in its monomer and is in fact insoluble in all of the commoner organic solvents. It is also worth mentioning that, on cracking, polyacrylonitrile, unlike some acrylic derivatives and linear polymers, does not cleave to give its monomer in any appreciable quantity. In this connection, an interesting study of the thermal degradation of some addition polymers has been made recently by Grassie [18].

The molecule of polyacrylonitrile is not so regular in shape as those of nylon and Terylene; also, it crystallises much less readily. Moreover, in the nitrile group the nitrogen atom is more electronegative than the carbon atom, and this results in a displacement of electron density towards the nitrogen atom, producing a high dipole moment. The dipoles of adjacent nitrile groups will thus interact, producing appreciable electrostatic forces of attraction between them. The effect of these electrostatic forces is accentuated by the close packing of the polymer chains, and is sufficiently large to make polyacrylonitrile difficult to melt and insoluble in the commoner solvents.

There are, however, a number of solvents which will dissolve polyacrylonitrile. These substances are, as might be expected, all highly polar in character, and include dimethylformamide, ethylene carbonate, methylene glycollate, propiolactone, butyrolactone, α-pyrrolidone, ε-caprolactam, and dimethyl sulphone. In addition, strong aqueous solutions of certain inorganic salts such as those at the end of the Hofmeister or lyotropic series, e.g. bromides, iodides, and thiocyanates, will dissolve polyacrylonitrile. In this connection, lithium bromide and sodium thiocyanate have proved of particular value.

Polyacrylonitrile may be dry- or wet-spun into yarns from its solutions in dimethylformamide. At the present time, however, fibres of 100% polyacrylonitrile are rarely used commercially because they are usually difficult to spin (owing to technical problems associated with the production of satisfactory spinning solutions) but mainly because they are extremely difficult to dye. Nevertheless, these fibres have excellent resistance to acids and chemicals generally. They have also excellent flexing properties, and exhibit outstanding resistance to photodegradation and abrasion. In filament form the yarns have a silk-like handle, while in staple-fibre form they have the handle and warmth of wool.

In order to overcome the inherent disadvantages of 100% polyacrylonitrile fibres it is necessary to copolymerise acrylonitrile with vinyl derivatives of a more polar character such as methyl acrylate, vinyl acetate, vinylpyridine, and vinylpicoline. Fibres produced from binary and ternary polymers of acrylontirile and these polar monomers are invariably more soluble and more easily dyed than those made from polyacrylonitrile alone. The more open structure of the copolymers also accounts for their greater solubility, which facilitates spinning. It affords a greater number of dye sites, which together with the presence of the polar groups tend to increase the rate of absorption of dyes.

For an acrylic fibre to have good tenacity it must contain at least 80% acrylonitrile. Usually, however, the currently available acrylic fibres contain about 90% acrylonitrile. Copolymers containing 90% acrylonitrile can be readily prepared in a suitable solvent, and either dry- or wet-spun as desired.

Although the fibres produced from these copolymers are less resistant to heat, chemical attack, flexing, photodegradation, and abrasion than those produced from 100% polyacrylonitrile,

they are nevertheless commercially very satisfactory with regard to these properties. The tensile strength of the fibres is, on the other hand, very little different from that of 100% polyacrylonitrile fibres.

As the percentage of acrylonitrile in the copolymers decreases, their solubility in the commoner organic solvents increases. For example, fibres produced from a copolymer containing 70% of acrylonitrile and 30% (by weight) of a more polar vinyl monomer are soluble in acetone and have a moisture regain of 6–7%, which is very similar to that of yarns of secondary cellulose acetate. Indeed, the American semi-acrylic fibre dynel (Union Carbide & Carbon Corpn.), which contains 40% acrylonitrile and 60% vinyl chloride (by weight), is soluble in acetone and may be dry-spun from this solvent.

6.1. *Properties and Applications of the Acrylic Fibres*

An outstanding and very desirable property of the acrylic fibres is their resistance to outdoor exposure in general and to sunlight in particular.

The acrylic fibres have a warm, wool-like handle, good resilience, good resistance to abrasion, good resistance to chemical attack as well as to micro-organisms and insects, and good bulking power. Undoubtedly, the most important property and one which is characteristic of the acrylic fibres is their ability to be stretched, producing high-shrinkage fibres which can be used to yield high-bulk yarns.

It is well known that for every high polymer there is a narrow temperature region at which it characteristically changes from a more or less brittle state to a rubbery or viscous state. This temperature region is called the "glass temperature"[13] or *second-order transition temperature*.

Owing to the chemical architecture and flexibility of the polyacrylonitrile chains in the acrylic fibres, if these are heated above about 60°c. (the second-order transition temperature), stretched, and then cooled under tension, they will, on reheating, contract to their original length. If acrylic fibres which have been set in this way are then blended with acrylic fibres which have not been so treated, the resulting yarn becomes wool-like when heated as a result of the unstretched fibres being crimped by contraction of the set fibres. The process is known as *bulking*, and the yarns are said to be *bulky*. Generally, however, they are referred to as *high-bulk yarns*.

High-bulk acrylic yarns have been widely applied commercially for the production of knitted garments such as jumpers, twin-sets, and sweaters. These have the appearance of similar garments knitted from woollen yarns. Because of their lightness combined with warmth, these garments have naturally met with wide consumer approval.

Yarns produced by blending the acrylic fibres with the natural fibres such as wool and cotton and the man-made fibres such as viscose rayon staple fibres have found many worthwhile applications. For example, blends with wool have been successfully used in ladies' and men's suitings, the main attraction of these blends being that they combine warmth with lightness and good wearing properties.

Blend yarns of acrylic fibres and cotton or viscose rayon staple fibres are now being vigorously developed, especially in this country and in the U.S.A. The great desire of housewives for quick-drying garments, which require little or no ironing after machine-washing, has been claimed as the main reason for their rapid development. Blends of 80% acrylic fibre plus 20% cotton and 70% acrylic fibre plus 30% viscose rayon staple fibre have, for example, been recommended for the production of men's shirts and ladies' blouses, and it is claimed that materials produced from these yarns have good wash-and-wear properties, excellent aesthetic appeal, an agreeable, soft handle, and high abrasion resistance.

Finally, a brief reference should be made to the processing and dyeing of yarns of the acrylic fibres and blends of these with wool, cotton, or viscose rayon staple fibres. All of these yarns may be processed satisfactorily on conventional textile machinery. In sizing warps of yarns based on 100% acrylic fibres, sizes consisting of thin-boiling modified starches, polyvinyl alcohol, and a lubricant (such as tallow or a water-dispersible wax) offer adequate protection to them during weaving. As the high-bulk acrylic yarns shrink at about 60°c., care must be taken to ensure that the temperature of the size during its application to the yarns as well as the temperature of the drying cylinders is kept below 60°c. In sizing ordinary acrylic yarns, the temperatures employed are not so important; nevertheless, it is advisable to dry at as low a temperature as is possible. In all cases overstretching during sizing should be avoided, 1·5% being considered the maximum desirable.

The importance of temperature in the sizing of blend yarns containing high-bulk acrylic yarns must again be stressed. Generally, a size which is satisfactory for warps of ordinary or high-bulk acrylic yarns is also satisfactory for warps of blend yarns containing a high percentage (say 70%) of the acrylic fibre. For blends in which the percentage of the acrylic fibre is under 50%, size mixtures which are considered satisfactory for the major component of the blend are usually suitable.

The acrylic fibres can be dyed as yarn or fabric in conventional dyeing equipment at the boil under normal atmospheric pressure. They are most readily dyed with basic dyes and with disperse dyes.

Basic dyes are usually applied from an acid bath at the boil. Some of the newer types of basic dyes, e.g. the Sevrons (DuP), Deorlenes (Ciba), and Maxilons (Gy), give dyeings of very good light fastness on acrylic yarns.

Disperse dyes are applied to the acrylic yarns from a soap solution as direct dyes but at higher temperatures. Only pale and medium depths can be obtained satisfactorily. With proper selection of dyes, however, high fastness to light can be obtained.

The dyeing of blends of the acrylic fibres with other fibres offers no real difficulties. For example, an acrylic–wool blend may be dyed in two stages

from one bath: the wool is dyed first, then the pH is adjusted, and the acrylic is dyed. An acrylic–viscose rayon staple blend is dyed by first dyeing the acrylic and then dyeing the viscose rayon in a second bath.

7. CONCLUSIONS

It is clear from what has already been said about acrylonitrile that it has made a considerable impact on the textile industry.

With so many of the large chemical companies of the world now manufacturing acrylonitrile, it is certain that a vast amount of research and effort is being devoted to the discovery and development of new textile applications for this extremely reactive and valuable organic chemical.

Production of acrylonitrile in the U.S.A. has grown from 57 million lb. in 1953 to 141 million lb. in 1956. Figures for the world production of acrylic fibres are more difficult to obtain, but it would appear that in 1955 production of these fibres was about 60 million lb., and in 1956 it had risen to about 85 million lb.

These figures clearly emphasise the tremendous development of acrylonitrile which has occurred within recent years. They also indicate the vast growth potential of this substance, which may well prove to be the most outstanding and versatile industrial organic chemical of the second half of the twentieth century.

* * *

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Discussion

Mr. J. A. Bates: Would it be true to say that the greater the degree of shrinkage of the bulking yarn the more effective the final blend becomes, i.e. the more wool-like? If this were so, the copolymers with higher shrinkage would be most effective.

Dr. MacGregor: Although it is necessary that there should be a large differential shrinkage between the two components in a yarn to produce bulking, this is not the only factor which must be considered. In the first place there must be a sufficiently high degree of interaction between the two components, which, of course, is achieved through frictional forces, and secondly the tension–shrinkage relationship of the high-stretch component is of importance. In view of these additional factors, it does not necessarily follow that the greater the free shrinkage of the stretched component the greater is the bulking capacity of the yarn.

Mr. H. R. Hadfield: The change in physical properties above the second-order transition point can be a source of serious difficulty to the dyer and finisher. Can the second-order transition point of polyacrylonitrile fibres be raised by chemical modification?

Dr. MacGregor: It was expected that the change in physical properties of polyacrylonitrile fibre above the second-order transition temperature would be a source of difficulty to the dyer and finisher. However, no difficulty has been encountered in practice. Although it may be possible to raise the second-order transition temperature by chemical means, this would very likely reduce the advantages of copolymerisation.

Dr. L. Valentine: With regard to Mr. Hadfield's question, copolymerisation can modify the second-order transition temperature, but it would surely be more likely to depress the transition temperature of polyacrylonitrile than to elevate it.

Dr. MacGregor: Whether or not the second-order transition temperature is lowered by copolymerisation depends upon the nature of the substituent. If this is such as to increase the flexibility of the chain, then the second-order transition temperature will be depressed; if it serves to stiffen the chain, then the transition point will be raised. In the majority of cases with polyacrylonitrile the substituent lowers the second-order transition temperature. Thus the second-order transition temperature of pure polyacrylonitrile is above 100°C., whereas that of commercial copolymers is in the range 60–70°C.

Mr. L. S. Newton: What precautions are necessary in the sizing of yarns containing high-bulk fibres?

Dr. MacGregor: Owing to the fact that high-bulk acrylic yarns shrink at temperatures of about 60°C. it is essential to ensure that the temperatures of the size-bath and of the drying cylinders are kept below this value during the sizing operation. Sizing stretch should be kept as low as possible and should not exceed 1·25%. By using nine drying cylinders it is possible to attain sizing speeds of 30 yd./min.

As already mentioned, the most satisfactory sizes appear to be those based on a thin-boiling starch (e.g. a starch ether), polyvinyl alcohol (which acts as a film strengthener and binder), and a water-dispersible wax. Such a size mixture can

be applied to the warp yarns at 50–60°C. In the case of warps of ordinary acrylic yarns, however, the size is usually applied at 70°C.

Mr. A. P. LOCKETT: Have higher saturated or unsaturated nitriles been used in similar reactions to those of acrylonitrile?

Dr. MACGREGOR: Unfortunately, none of the higher unsaturated nitriles are so reactive as acrylonitrile. Even α-methylacrylonitrile is not nearly so reactive chemically as acrylonitrile itself, although it may be easily polymerised. Unlike polyacrylonitrile, polymethacrylonitrile is soluble in the commoner organic solvents.

Many saturated nitriles, e.g. acetonitrile, are very satisfactory and useful solvents. Some of the higher saturated and unsaturated nitriles are useful lubricants, and many are the constituents of oils used in the lubrication of textile yarns.

The organic nitriles, as a class, are useful intermediate substances, and adiponitrile is, in fact, an important intermediate in the manufacture of nylon.

COMMUNICATION

The Supercontraction of Wool Fibres by Aromatic Compounds

W. G. CREWTHER

The kinetics of supercontraction of wool fibres have been investigated using solutions of phenol and other aromatic compounds over a large range of concentrations and pH. The evidence shows that the phenolate ion together with its associated cation is more effective than the un-ionised form of phenol in causing supercontraction of wool fibres. However, the un-ionised form of phenol also causes contraction, and is chiefly responsible for contraction of the wool at low pH values. It is suggested that the two forms of phenol cause contraction of wool fibres by different mechanisms.

Zahn [1] and Elöd and Zahn [2] have shown that horsehair, wool, other animal fibres, and polyamide fibres supercontract when heated in aqueous solutions of phenol at temperatures greater than 70°C. In addition horsehair was shown to contract when heated in solutions of *p*-nitrophenol or *m*-nitrophenol, but insufficient evidence was obtained to determine the mechanism of the change and whether the phenols were active in the ionised or un-ionised form.

Crammer and Neuberger [3] and Laskowski and Scheraga [4] have postulated the existence of relatively strong hydrogen bonds between un-ionised tyrosine side-chains and other side-chain groups of proteins. Phenols in their un-ionised form might be expected to compete with tyrosine residues and other side-chain or main-chain groups in the formation of such hydrogen bonds and so rupture the original bonds.

On the other hand, phenols are adsorbed at surfaces, by proteins in general, and by the wool fibre in particular [1]. If phenol is adsorbed on the wool as the anion, the net negative charge of the wool will be increased. This will cause swelling of the fibres as a result of electrostatic effects, the establishment of a Donnan equilibrium, and transport of water into the fibres with the associated cations [5] and so favour contraction. If this explanation is correct, the phenol is acting as a source of readily adsorbed anions, and the mechanism of supercontraction in phenol solutions may be similar to that in solutions of salts such as iodides. The similarities between the action on wool fibres of solutions of phenol and concentrated salt solutions—supercontraction without appreciable changes in disulphide content [1, 6, 7] and dissolution of a portion of the cortex of the fibre by the reagent [1, 7]—are readily explained on this basis.

It is therefore important to assess the relative effects of the ionic and non-ionic forms of phenol. This paper describes experiments which provide details of the kinetics of supercontraction of wool fibres in solutions of phenols and other aromatic compounds at various pH values and concentrations. Histological changes at various stages of contraction are also described.

MATERIALS AND METHODS

Wool

Corriedale 56s wool from a uniform fleece was cleaned by extraction with cold light petroleum (b.p. 40–60°C.), once with cold ethanol, and several times with distilled water; the wool was dried in a stream of air at 40°C.

Reagents

The phenol used was of B.P. standard (Evans); its purity was such that redistillation increased the rate of contraction of wool fibres in solutions of phenol only slightly (cf. Zahn [8]). Other organic chemicals were of laboratory reagent quality (B.D.H.), and these were further purified by recrystallisation if it was considered necessary. Formic, acetic, and boric acids and salts used for buffering were of analytical reagent quality.

Procedure

Single fibres were mounted in glass tubes (6 mm. in diameter × 150 mm. long) by attaching each with softened polystyrene to a short fine glass rod inserted in a rubber stopper closing the lower end of the tube and in a similar manner to a long fine glass rod passing through a bark stopper closing the upper end of the tube. A white-on-black scale was attached to the tube with rubber bands. The tube was placed in a test-tube of water, which was maintained at a temperature of approx. 98·5°C. by immersion in a boiling water bath. The fibre was slack during the process of heating, but in order to measure the length of the fibre on the scale the glass rod was moved upwards until the

crimp was just removed; after the measurement the fibre was again slackened. An accuracy of $\pm$ 0·5% of the original length of the fibre was attainable. Solutions to be tested were heated in the bath before being poured into the tube, and length measurements were made at suitable time intervals. The pH of the test solution at a temperature of about 60°C. was measured before and after heating with the fibre by means of a glass electrode (pH 1–11) and electrometer (Jones, Melbourne).

In some experiments the amount of protein passing into solution from the fibres during supercontraction was determined by placing 200-mg. samples of conditioned wool in flasks containing 250 ml. of the phenol solution at 98·5°C. The wool was opened out in the solution and allowed to digest for periods of 10–240 min., washed three times by transferring to 50-ml. aliquots of distilled water and allowing to stand for 1 hr., and dried, conditioned, and weighed. The residues were assayed for total sulphur by Dr. K. W. Zimmermann of the C.S.I.R.O. Microanalytical Laboratory. The kinetics of contraction of fibres under comparable conditions of liquor ratio and concentration using 20 mg. wool and 25 ml. solution were shown to be similar to those observed under the standard conditions; the apparatus described by Crewther and Dowling [7] was used for this purpose.

RESULTS

Kinetics of Contraction in Phenol Solutions of Different Concentration and pH

Measurements were made of the changes in length of fibres while heated at 98·5°C. in 0·5, 1·0, or 2·0 M. phenol solutions at pH values varying from negative values (2 or 5 N-HCl) to 10·5. For pH values greater than 2·0 the solutions were buffered with 0·1 M. solutions of acids or bases adjusted to the desired pH with NaOH or HCl—pH $<$ 4 formate, 4–6 acetate, 6–7 succinate, 7–8 trishydroxymethylaminomethane, 8–10 borate, $>$ 10 piperidine. Where very low or very high pH values were used, fibres were also tested in solutions of equal acid concentration or of equal pH. The rate curves are of three types—those in which there is an immediate rapid contraction to a minimum length followed by a more or less rapid extension to a length which may be even greater than that of the original fibre (Fig. 1 and 8); those in which the fibre contracts to an intermediate level, at which the length remains almost constant for a period, followed by a second stage of contraction to a minimum length and in some cases subsequent elongation (Fig. 1, 7, and 8); and those in which there is an initial lag period followed by contraction to a minimum length with subsequent extension (Fig. 7 and 8). Some curves of the last type show also a two-stage contraction.

Fig. 2 shows the extent of supercontraction after 30 min. as a function of the pH of 1·0 and 2·0 M. solutions of phenol. With 1·0 M. phenol there is practically no contraction at pH values below 5·0 apart from that in 2 N. or 5 N. hydrochloric acid, and in the absence of phenol such acid solutions produce effects approximating those in acid

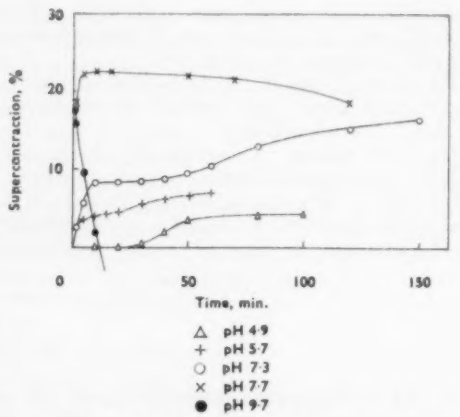


FIG. 1—Kinetics of Contraction of Wool Fibres in 2·0 M. Solutions of Redistilled Phenol at Various pH Values

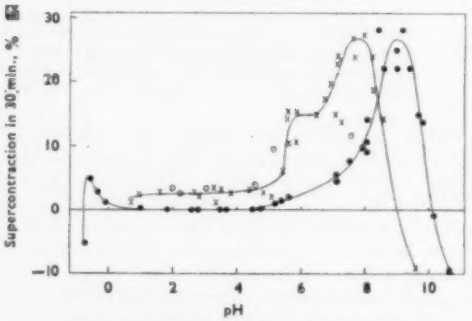


FIG. 2—Relation between pH of Phenol Solutions and Supercontraction of Wool Fibres heated in the Solutions for 30 min.

solutions of phenol. As the pH is increased above 6·0 the extent of contraction increases very greatly to a maximum value at about pH 8·4. The reduction in contraction with further increase in pH or when highly acid solutions are used is a result of the rapid extension of fibres which follows contraction in alkaline solutions or in solutions containing 2 N. or 5 N. hydrochloric acid. With 2·0 M. solutions of phenol a similar general pattern is obtained except that the maximum value occurs at a lower pH (7·8) and that there is a small but definite contraction at pH values below 5·0. The flattening of the curve near pH 6 is a reflection of the two stages in the contraction kinetics.

Considerable contractions are obtained in the range of initial pH values 0–6 when unbuffered solutions of 1·0 M. phenol are used and the contraction is allowed to proceed for an extended period (Fig. 2). This is partly due to a steady increase in pH of the unbuffered phenol solutions while being heated with the wool fibres, but the ionic strength in buffered solutions may have a small inhibitory effect on the contractions.

The maximum contraction obtained varies with the pH, the maximum value being least at the extremes of pH and greatest at intermediate

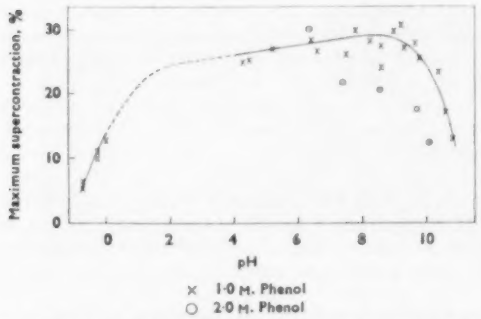


FIG. 3—Relation between pH and Maximum Contraction of Wool Fibres in Phenol Solutions

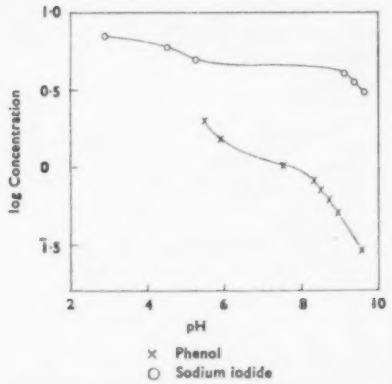


FIG. 4—Comparison of the Effect of pH on the Concentration of Phenol and Sodium Iodide causing 10% Contraction of Wool Fibres in 1 hr. at 98·5°C.

values (Fig. 3); with 2·0 M. solutions of phenol the maximum values in the alkaline range are less than those obtained with 1·0 M. solutions (Fig. 3).

Having determined the kinetics of contraction at different pH values and phenol concentrations, it is possible by interpolation to determine for each pH value the concentration of phenol required to cause a predetermined contraction in a fixed interval of time. This relationship is compared with similar data from contractions in solutions of sodium iodide [7] in Fig. 4. As the pH increases over the range where ionisation of phenol would be expected, the concentration of phenol required is reduced more than that of sodium iodide over the same pH range. With both sodium iodide and phenol the concentration required decreases very greatly as the pH is increased above about 8·0.

In order to determine whether diffusion of the phenol into the fibres limits the rate of contraction, fibres have been soaked in 0·5 M. phenol solutions at pH values ranging from 8·0 to 9·5 for 20 hr. at 2°C. before heating in the usual manner. There is no marked change in the lag period or the shape of the curves relating contraction and time; the contraction is generally slightly slower than with unsoaked fibres. No measurable contraction occurs during soaking.

Changes in the Appearance and Histology of the Fibre during Supercontraction

The orthocortex contracts earlier than the paracortex, and this results in straightening of the fibre followed by intense curling. Under conditions which cause rapid contraction of the fibre these changes are not always apparent, but, in solutions which require an initial lag period before contraction of the paracortex becomes measurable, straightening and intense curling of the fibre are invariably observed. The subsequent contraction of the paracortex is accompanied by restraightening of the fibre and corrugation of the cuticle across the fibre (Fig. 5A), and this effect is most pronounced when maximum contraction is reached. Further heating, particularly in alkaline or concentrated solutions of phenol, causes the fibre to extend (Fig. 1 and 8) and the corrugations to become less conspicuous. They can be faintly observed, but only by using phase contrast, when the original length has been reached. During this process of elongation the fibre again curls, and later straightens as the original length is exceeded. All length measurements in these experiments have been made after applying sufficient tension to remove crimp from the fibres. Consequently, the paracortex is initially under slight strain until contraction of the orthocortex begins; thereafter length changes refer to the more resistant paracortex of the fibre.

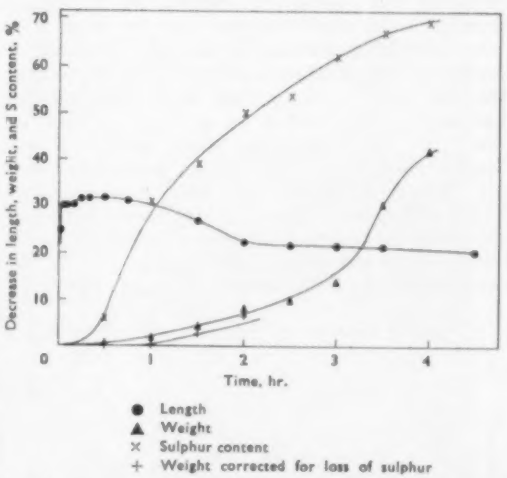


FIG. 6—Changes in Weight, Sulphur Content, and Length of Wool Fibre with Time of Heating in 1·0 M. Phenol at pH 9·4

In 1·0 M. phenol at pH 9·4 maximum contraction is reached in about 30 min., and elongation takes place at first rapidly and then more slowly. Fig. 6 compares changes in length, weight, and sulphur content of wool heated in 1·0 M. phenol at pH 9·4 using a liquor ratio of 1250:1. If the wool is recovered and washed with continual tamping on a Buchner funnel, the amount passing into solution is considerably greater than that shown in Fig. 6 and may reach 70%. During the heating process the fibre swelling increases, particularly in the orthocortex, and this segment tends to lose the

longitudinal striations which characterise the paracortex and develops a granulation (Fig. 5B). At this stage (2 hr.) some fibres also develop longitudinal cracks in the cuticle, which may result from swelling of the cortex. After 4 hr. the residues consist of cell nuclei with resistant membranes and fibrillar material surrounded by a cuticular remnant which still shows scale markings (Fig. 5C and 5D). The cuticle tears easily, and often some of the cortex, which has little lateral cohesion, adheres to the cuticle.

With 2·0 M. phenol at pH 9·4 these changes take place more quickly. Maximum contraction occurs in less than 2 min., and elongation to a length greater than the original follows rapidly. Dissolution of the fibre is also more rapid and extensive, particularly from the orthocortex. After heating for 3 hr. at 98·5°C. the residue consists of a flat tube of cuticle (Fig. 5E) containing nuclei and resistant fibrillar and membranous material (Fig. 5F). The cuticular remnant shows little structure unless phase-contrast microscopy is used, when scale markings and longitudinal striations, probably due to adhering cortex, are clearly visible (Fig. 5G). These residues resemble closely those obtained by treating fibres with peracetic acid and extracting with aqueous ammonia [9].

Supercontraction with Compounds related to Phenol

A number of substituted phenols and other aromatic compounds were tested by the usual procedure, the concentration used for each test compound being limited by its solubility and a higher pH being used for the less soluble substances. The less soluble compounds and some soluble compounds were tested at a concentration of 0·25 M. at pH 10 in 0·1 M. borate buffer. These compounds included *o*-, *m*-, and *p*-cresol, *o*-, *m*-, and *p*-methoxyphenol, *m*- and *p*-nitrophenol, 2:4:6-trinitrophenol (picric acid), benzene-1:2-diol (catechol), benzene-1:3:5-triol (phloroglucinol), 3:4:5-trihydroxybenzoic acid (gallic acid), 5-methylbenzene-1:3-diol (orcinol), and disodium phenyl phosphate. The kinetics of contraction of fibres heated in these solutions were compared with those of contractions in phenol solutions under similar conditions of concentration and pH.

In phenol the fibres curled tightly after about 5 min., but no contraction of the paracortex took place even after heating for 270 min. Similar results were obtained with catechol, phloroglucinol, and gallic acid; the kinetics of contraction of fibres in the other reagents are shown in Fig. 7. It is apparent that substitution of a methyl or methoxy group for a hydrogen atom on the benzene ring as in the cresols, orcinol, and methoxyphenol increases the tendency for the fibres to contract. Both with the cresols and with the methoxy compounds the position of the substitution is not important. Nitration of phenol also increases the rate of contraction, the monosubstituted compounds being more effective than the trisubstituted compound.

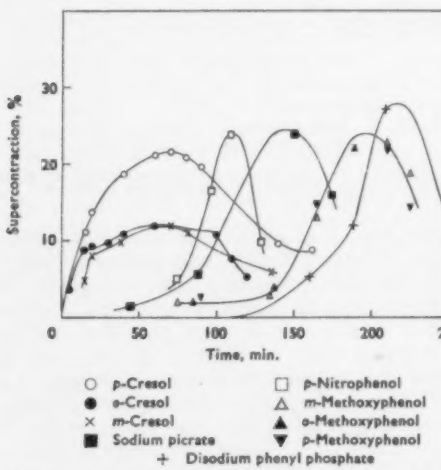


FIG. 7—Kinetics of Contraction of Wool Fibres in 0·25 M. Solutions containing 0·1 M. Sodium Borate at pH 10·0

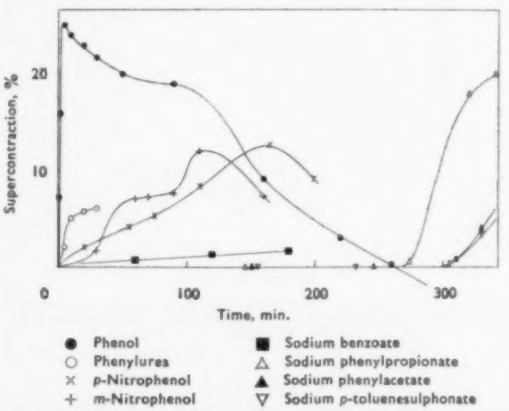
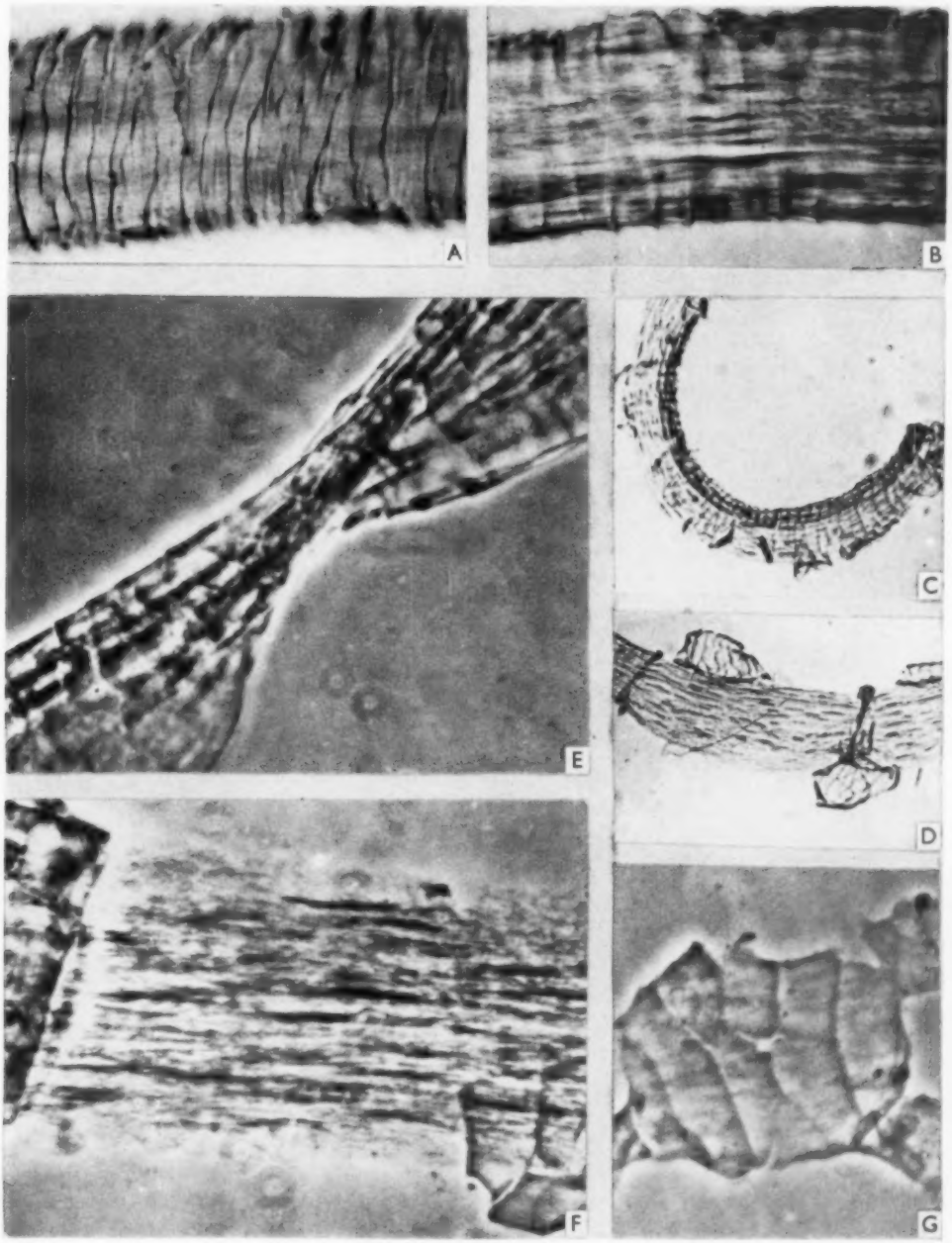


FIG. 8—Kinetics of Contraction of Wool Fibres in 1·0 M. Solutions containing 0·1 M. Sodium Borate at pH 9·0

The more soluble benzene derivatives were tested in 1·0 M. solution at pH 9·0 using 0·1 M. borate as buffer. These compounds included—disodium phenyl phosphate, *m*- and *p*-nitrophenol, phenylsemicarbazide, phenylurea, sodium *p*-toluenesulphonate, sodium phenylacetate, sodium phenylpropionate, and, for comparison with an aliphatic compound, sodium decanoate. Fig. 8 shows that under these conditions phenol was much more effective than the other compounds, the rate of contraction decreasing in the order—phenylurea > *p*- and *m*-nitrophenols > benzoate > phenylpropionate > phenylacetate > *p*-toluenesulphonate > decanoate. In view of the anomaly in the relative effect of phenol and *p*- and *m*-nitrophenol which is apparent on comparing Fig. 7 and 8, further experiments were carried out with these compounds and phenol using various concentrations at pH values approximating 10. The increase in the rate of contraction of the fibres on increasing the concentration of the reagent was much greater with phenol than with the nitrophenols. The anomaly is therefore related to the concentration of the phenols, not to the pH of the solutions.



(A) 1·0 M. for 40 min. (× 600)
(B) 1·0 M. for 2 hr. (× 600)
(C) and (D) 1·0 M. for 4 hr. (× 150)
(E) 2·0 M. for 3 hr. showing flattening of the fibre (phase contrast, × 600)
(F) 2·0 M. for 3 hr. showing residual fibrillar material between breaks in the cuticle (phase contrast, × 600)
(G) Cuticular remnant from a fibre heated in 2·0 M. phenol for 3 hr. (phase contrast, × 600)

FIG. 5—Corriedale Fibres heated at 98·5°C. with Solutions of Phenol at pH 9·4

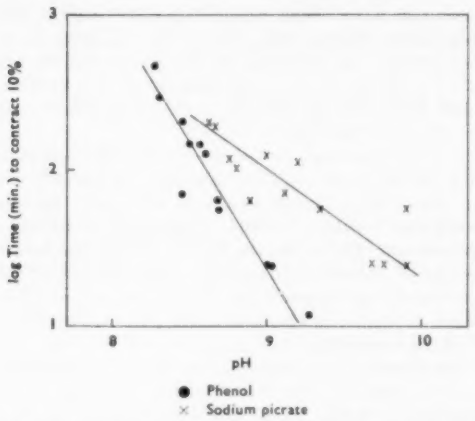


FIG. 9— Comparison of the Effect of pH on the Time for 10% Contraction in 0·5 M. Solutions of Phenol and Sodium Picrate

A comparison was made of the kinetics of contraction of wool fibres at various pH values in 0·5 M. phenol and in 0·5 M. picrate buffered with 0·1 M. borate or piperidine. The results are expressed as a relationship between the pH and the time required for the fibres to contract 10% (Fig. 9). As the pH is increased, the acceleration in contraction is greater in solutions of phenol than in solutions of picrate.

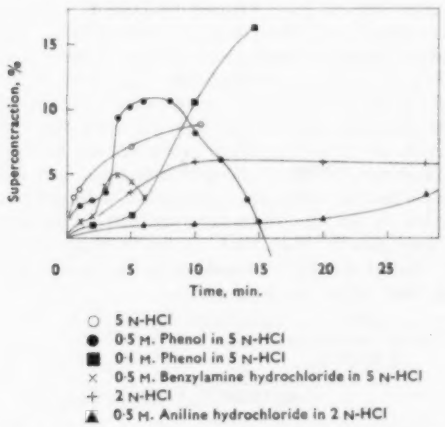


FIG. 10— Kinetics of Contraction of Wool Fibres in Acid Solutions of Aromatic Compounds

Contraction of wool fibres in solutions of benzylamine and aniline hydrochloride has been investigated. Like phenol, these compounds have little effect on the rate of contraction in 5 N. or 2 N. hydrochloric acid (Fig. 10), but benzylamine, like phenol, increases the rate of the elongation which follows contraction. At pH 10·3, 0·5 M. benzylamine, with about 90% of the amine in the un-ionised form, causes the wool to contract at a rate comparable with that obtained with 0·5 M. phenol at pH 9·2. It is noteworthy that the maximum contraction which can be obtained with 0·5 M. phenol in 5 N. hydrochloric acid is small compared with that in 0·1 M. phenol in 5 N. hydrochloric acid (Fig. 10). The more dilute phenol solution also gives a much greater maximum contraction than 5 N. hydrochloric acid containing no phenol.

DISCUSSION

This investigation has provided the following evidence that the phenolate ion is chiefly responsible for the thermal contraction of wool fibres in hot alkaline solutions of phenol—

(1) The rate of contraction increases markedly with pH at values approaching the pK_a of phenol (Fig. 2).

(2) The concentration of phenol required to cause 10% contraction of wool fibres after heating for 60 min. decreases sharply with increasing pH over the range of values in which phenol is converted to phenolate ions (Fig. 4). Although with increasing pH there is also a decrease in the concentration of sodium iodide required to produce a contraction of this magnitude under similar conditions, the variation in concentration of the sodium iodide is proportionately much smaller.

(3) The rate of contraction of wool fibres in alkaline solutions of sodium picrate is comparable with that in phenol solutions of the same concentration and pH (Fig. 9). Since the pK value of picric acid is $< 1{\cdot}0$, the effective concentration of un-ionised picric acid in a 0·5 M. solution at pH 10 would be of the order of 10^{-10} M. Similarly, *p*-nitrophenol with a pK of 7·14 and *m*-nitrophenol with a pK of 8·35 are more effective than phenol (pK 9·95)[10] in alkaline solutions at low concentrations (Fig. 7 and 9). It is therefore unlikely that the un-ionised forms of picric acid or *p*-nitrophenol play any part in inducing contraction of wool fibres at pH 10.

(4) The increase in the rate of contraction in alkaline solutions with increasing pH is much less with sodium picrate than with phenol (Fig. 9). Since the ionisation of picric acid is almost unchanged over the experimental pH range, this result would be expected only if the ionised forms of picrate and phenol are mainly responsible for the contraction.

Although on a molar basis the ionic form of phenol is more effective than the un-ionised form, this simple explanation cannot account quantitatively for the results in Fig. 4. At pH 5·6, 2 M. phenol, in which the concentration of phenolate ions would be approx. 1×10^{-4} M., causes wool to contract at about the same rate as 0·5 M. phenol at pH 9·1, in which the phenolate concentration would be about 0·1 M. The rate of contraction in solutions of sodium iodide and picrate, in which ionisation is not appreciably affected by pH, increases with increasing pH probably as a result of increasing lability of –S·S– cross-linkages. It follows that at least one component of the system other than phenolate ion must facilitate contraction in solutions of phenol. This is probably a direct effect of un-ionised phenol molecules on the contraction. The presence of positive charge on the hydroxy group of the phenol due to resonance with the benzene ring would permit the formation of relatively strong hydrogen bonds with carboxyl or similar acceptor groups[4]. As the concentration of phenol relative to that of water was raised, it would tend to displace the water and compete

with protein side-chain groups for hydrogen-bonding sites and so favour thermal disordering of the fibre. This is also demonstrated by the contraction of wool fibres caused by 2·0 M. solutions of phenol at pH values of 2–3 (Fig. 2), i.e. with concentrations of phenolate ions of less than 2×10^{-7} M. The curve for phenol in Fig. 4 also shows a greater effect of pH in the range 5–8 than would be expected solely from a consideration of the ionisation of phenol. More information concerning the effects of temperature and phenol concentration on the pK values of phenol and side-chain groups of proteins is required for the interpretation of these effects.

Other factors which may influence the rate of contraction are the rate of formation of lanthionine (cf. Fig. 6) and the ionic strength of the solutions. The former may be influenced by the rate of disordering of the protein in relation to the pH value. The ionic strength would increase greatly with pH in solutions of phenol, whereas in solutions of sodium iodide or picrate the change in ionic strength with pH would be relatively small. The greater increase in rate of contraction with increasing concentration of phenol as compared with *p*-nitrophenol (Fig. 7 and 8) may also be due to the greater increase in ionic strength on increasing the concentration of nitrophenol, which is largely ionised at the experimental pH.

The conclusion that sodium phenolate is chiefly responsible for the contraction of wool fibres in alkaline solutions of phenol relates this work closely with observations of the contraction of wool and collagen in solutions of salts [5, 6, 11]. Most of the theories postulated to explain the disordering effect of salts are equally applicable to the effects of sodium phenolate. However, it is difficult to explain why benzoate and other aromatic carboxylic acids have considerably less effect than phenol at the same pH. More data concerning the hydration and adsorption of these ions are necessary before the results can be considered in terms of the theories postulated to explain the action of salts and their relation to the lyotropic series [5, 11].

The greater effect of the cresols and the methoxy derivatives of phenol as compared with phenol in alkaline solution (Fig. 7) may be due to the increased size of the hydrophobic part of the molecule. This could increase adsorption at the protein–solution interface and, on the basis of the theory proposed to explain the contraction of collagen by salts [5], this could increase the rate of contraction. From this viewpoint polyhydric phenols and compounds such as gallic acid should be less effective than phenol. The greater effect of nitrated phenols as compared with phenol (Fig. 7) can be explained in terms of the increase in ionisation of the molecule caused by nitration. It seems unlikely that the picrate ion would break hydrogen bonds by direct competition with the side-chain groups, since the negative charge would be largely distributed among the three nitro groups, and no single oxygen atom is likely to carry a large negative charge.

Extended lag periods before contraction is apparent have been observed only under sub-optimal conditions (Fig. 1 and 7). It seems likely that hydrolytic splitting of disulphide bonds and some preliminary disorganisation of the proteins takes place during this period and so predisposes the fibres to contract.

Zahn [1] has already reported the dissolution of wool proteins in alkaline solutions of phenol, and he also observed a marked decrease in the disulphide content of the residues as dissolution progressed. Fig. 6 shows that initially the loss in weight of the wool heated in 1·0 M. phenol at pH 9·4 can be attributed chiefly to loss of sulphur, which is probably eliminated during the formation of lanthionine in the alkaline phenol solutions. Subsequently the loss in sulphur must be attributed at least partly to the dissolution of a sulphur-rich fraction from the wool, since the sulphur content falls to less than half that of the original material. It has been shown [7] that in solutions of lithium iodide in 1 N. hydrochloric acid proteins are extracted from wool leaving a sulphur-rich residue; the difference in the results obtained under the different conditions of pH and supercontracting agent is probably related to the greater lability of the disulphide bonds as the pH is increased. Dissolution of protein material from the fibres commences soon after heating is begun (Fig. 6), and the period of dissolution corresponds with the period of elongation following the initial contraction. This suggests that the phenol solution causes a "loosening" of the protein structure, which initially permits contraction of the structure, and as the process continues the number of sites binding the units of protein together is decreased until slippage can occur and the restoring force exerted by uncontracted portions of the fibre tends to reverse the contraction process and cause elongation. In addition, the reduced longitudinal restriction on the fibre may permit longitudinal swelling greater than that normally observed, and this would permit elongation to lengths greater than that of the original fibre.

DIVISION OF PROTEIN CHEMISTRY
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VICTORIA
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(*Received 25th August 1958*)

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[1] Zahn, *Z. Naturforsch.*, **26**, 286 (1947); *Kolloid Z.*, **113**, 157 (1949).
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Scheraga, *Biochim. Biophys. Acta*, **23**, 196 (1957).
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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Fluorescent Brightening Agents in Surgical Dressings

Dr. Adams states that there appear to be no grounds for preventing the use of fluorescent brightening agents in surgical dressings [1]. Our Surgical Dressings Subcommittee believes there to be very good grounds for refusing to amend the *British Pharmaceutical Codex* so as to allow these agents to be present in the dressings described in this book. We have been unable to discover any objective data obtained by testing fluorescent brightening agents for toxicity in circumstances similar to those in which dressings are often used, i.e. prolonged contact with open wounds and grazes. In May 1957 I informed Dr. Adams that the committee would be very willing to consider evidence based on tests relevant to these circumstances, but to date we have not been given any such evidence. The multiplicity of fluorescent brightening agents described in the paper is itself a good reason for preventing their use until all types have been shown to be harmless; the alternative of devising specifications to identify and limit the agents used on each dressing seems impracticable.

Although fluorescent brightening agents may mask poor bleaching, they do not enhance appreciably the appearance of a dressing made from cloth which has been "well bleached to a good white". All or some of the agents may be as harmless as Dr. Adams believes but, until this has been confirmed by properly designed and relevant tests, it is unreasonable to expect those responsible for standards to accept the possibility of increasing the hazards to patients by permitting additives which serve so little purpose.

K. R. Capper
(*Editor, British Pharmaceutical Codex*)
The Pharmaceutical Society of Great Britain
17 Bloomsbury Square
London W.C.1

19th February 1959

[1] J.S.D.C., **75**, 22 (Jan. 1959).

I agree with Dr. Capper that multiplicity of fluorescent brightening agents makes it difficult to devise specifications to identify and limit their use in dressings. I admit also that long exposure to open wounds may be different from the general contact that would arise with textile materials treated with these agents either during manufacture or during laundering. My main purpose in referring to the action of the Pharmaceutical Society of Great Britain was to try to correct any misconceptions that might arise regarding possible toxicity of these compounds in their general use in textiles, paper, and detergents.

With regard to Dr. Capper's remarks that he was willing to consider any evidence that manufacturers could bring forward, I feel that his Committee is much more competent to arrange for such testing, and I offered in 1957 to supply samples for this purpose, together with their general chemical constitution. This I am still perfectly willing to do.

D. A. W. Adams
Hickson & Welch Ltd.
Castleford
Yorkshire

12th March 1959

Cellulose Substantivity

The recent paper by Giles and Hassan [1] provides further evidence of the important correlation between the extended conjugated system of π electrons in a dye and its substantivity towards cellulose. This is a very welcome contribution to our better understanding of this most complicated subject.

There are, however, two assumptions in this paper which call for comment—(*a*) the suggestion, in common with the views expressed by most other writers on this subject, that cellulose substantivity is due to van der Waals forces, and (*b*) the use, also in common with other writers on this subject, of affinity measurements for the purpose of correlating substantivity and constitution. It is the purpose of the present note to point out (*a*) that the arguments in favour of van der Waals forces are far from being convincing, and (*b*) that the use of affinity measurements in this connection may be definitely misleading.

van der Waals forces*

Most recent writers on the subject of cellulose substantivity have stressed the importance of the planar nature of the dye, because planarity enables the non-polar van der Waals forces to become fully operative by virtue of the close approach between dye and substrate. On the contrary, it must be admitted that flat dye molecules do not necessarily possess marked substantivity (e.g. haematoxylin is flat but possesses low substantivity), whereas substances which possess high substantivity, whether they be direct dyes, vat dyes, naphthols, or natural substances such as bixin, all possess the extended conjugated system of π electrons.

A further weakness of the argument is the fact that, although van der Waals forces should be operative with all atoms of a molecule, there is no increase in substantivity in a range of vat dyes where the molecular size is increased progressively by the addition of eight successive methylene groups [2]. Again, if planarity of dye and fibre is all that is required to account for substantivity, one would expect direct dyes to have a marked affinity for linear polyethylene. The suggestion has been put forward by Giles [3], on the basis of refractive index measurements and surface film experiments, that when cellulose is immersed in water, the hydroxyl groups are so firmly solvated

* In this note the phrases "van der Waals forces" and "non-polar van der Waals forces" have been used in the sense in which they have been used by other writers on this subject. In view of the very indefinite nature of the term "van der Waals forces", it would have been preferable to use the more exact terms *dispersion forces* or *London forces*.

that hydrogen bonding with dye could not possibly take place. This same argument could be used against the van der Waals force theory, for these forces fall off very rapidly with distance, and therefore if the surfaces of dye and fibre are separated by even a single layer of water molecules, the van der Waals forces would be reduced to a negligible value. Since these forces are inversely proportional to the seventh power of the distance, an increase in separation from, say, 1·5 A. to 3 A. would reduce the force to approx. 1% of its former value.

Considering all the available evidence, it would appear that the possibility of interaction between the hydroxyl groups of cellulose and the π electron system of the dye offers the most logical interpretation of cellulose substantivity. On this basis, the mechanism could be explained as follows. In liquid water the molecules are arranged so that as much intermolecular hydrogen bonding as possible occurs. The arrangement is not as regular as in ice, but there will be as high a degree of local regularity as the thermal vibrations, etc. will allow. The average repeat distance of the water molecules will be quite different from the repeat distance between the hydroxyl groups of the cellulose. Therefore, in the neighbourhood of the cellulose surface the regular arrangement of water molecules will be disturbed, and fewer hydrogen bonds will be formed than in the bulk of the liquid. This results in a loss of stability and an increase in energy. It also explains the apparent paradox that, even if cellulose is too firmly bonded to water molecules to form hydrogen bonds with dyes or similar compounds, it can, nevertheless, form hydrogen bonds with the correctly spaced hydroxyl groups of an adjacent cellulose chain; for this is what happens when cellulose is precipitated from aqueous solution. On the other hand, with substances such as glucose and cellobiose, the water molecules can apparently arrange themselves more favourably round the hydroxyl groups of the much shorter molecule, and these substances, as distinct from cellulose, are therefore soluble in water.

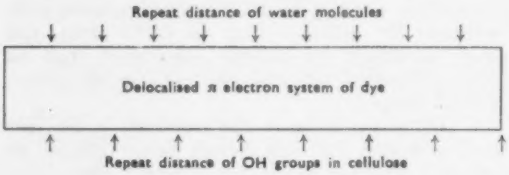

FIG. 1

The extended π electron system of the dye molecule is an ideal arrangement to form an intermediate layer between the cellulose on the one side and the regularly arranged water molecules on the other, since interaction can take place between the delocalised π electrons and the hydroxyl groups on either side, without any restriction as to spacing (Fig. 1). The same mechanism would apply even if the cellulose hydroxyl groups were solvated.

AFFINITY MEASUREMENTS

When a dye is adsorbed by the fibre, the heat of dyeing represents the energy of "bond formation" between dye and substrate. Entropy changes also occur during dyeing, owing to "restriction in mobility" imposed during adsorption. Affinity is related to these two by the well known equation—

$$-\Delta G^\circ = -\Delta H^\circ - T\Delta S^\circ$$

and is therefore merely the difference between two opposing factors, one of which is temperature-dependent. The affinity, therefore, is not a constant for any dye–fibre pair, but varies with temperature. Moreover, the way in which affinity varies with temperature differs considerably from dye to dye; e.g. at 60°C. the affinity of Durazol Fast Yellow 6G (C.I. Direct Yellow 46) for cellulose is greater than that of Chlorazol Fast Scarlet 8B (C.I. Direct Red 26), whereas at 90°C. the order is reversed[4]. It is therefore quite useless to expect any exact relationship to exist between affinity and constitution.

Whenever the affinities of a series of related compounds are compared over a range of temperatures, these reversal effects are found. The azoic coupling components offer a most interesting example: at 15°C. Naphtol AS–OL (C.I. 37530) has a higher affinity than Naphtol AS (C.I. 37505), but at 90°C. the order is reversed; thus *ortho* substitution with a methoxy group can increase affinity at one temperature and reduce it at another. It is only when we have resolved affinity into its conflicting components— heat of dyeing and entropy of dyeing— that we begin to obtain a true picture of the energetics of the dyeing process.

W. L. LEAD

TEXTILE DEPARTMENT
NOTTINGHAM AND DISTRICT TECHNICAL COLLEGE

16th February 1959

[1] Giles and Hassan, J.S.D.C., **74**, 846 (1958).
[2] Peters R. H., and Sumner, *ibid.*, **71**, 130 (1955).
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[4] Marshall and Peters, R. H., J.S.D.C., **63**, 446 (1947).

Spacing of the Blue Light-fastness Standards

Mr. Friele's interesting letter[1] gives me the opportunity of recording an observation I made in 1954 which confirms his findings that spectral composition is not the sole factor affecting the spacing of standards. I exposed Standards 1–4 (*B.S.* 1006) to three different U.K. daylight conditions— winter, summer, dull days only, and summer, sunny days only. The percentage increase in reflectance at 600 mμ. was found to be as follows—

Standard	Winter	Dull Summer	Sunny Summer
1	174	88	50
2	62	26	24
3	24	21	18
4	6	6	6

I cannot add anything to the possible explanations given by Mr. Friele, and can only endorse his conclusion that a better understanding of fading processes is still needed.

K. MCLAREN

HEXAGON HOUSE
MANCHESTER 9

26th January 1959

[1] Friele, L. F. C., J.S.D.C., **75**, 44 (Jan. 1959).

Notes

Meetings of Council and Committees March

Council— 4th
Publications— 17th
Examinations Board— 10th
International Federation 1959 Congress—
Scientific and Technical Subcommittee— 13th.

Deaths

We regret to report the deaths of Mr. W. Mann, Mr. F. W. Rayner, and Dr. Luigi Sessa (*Immediate Past President of the International Federation of Associations of Textile Chemists and Colourists*).

Chemical Nomenclature

Changes of convention in chemical nomenclature have been adopted by the Chemical Society, and will be followed in the present and subsequent issues of this *Journal*. They affect mainly position numerals and the italicisation of prefixes, and are set out in full in *Proc. Chem. Soc.*, 4–5 (Jan. 1959). They are summarised below.

Numbers forming a single chemical series will be separated by commas, e.g. 1,2,4-trihydroxyanthraquinone, but the colon will be retained for separating sets of series of numbers, e.g. 1,2:7,8-dibenzanthracene.

Prefixes (other than numerals or symbols) in the names of chemical compounds or radicals will be italicised only when they define the positions of named substituents or define stereoisomers, e.g. *N*-methyl-*p*-nitroaniline, *trans*-stilbene. Other prefixes will be in roman type, and "secondary" and "tertiary" in the aliphatic series will now be abbreviated to "s-" and "t-" respectively, e.g. cyclohexane, *p*-t-butylaniline, isopropyl. These changes will affect the alphabetical order in indexing and in naming substituents in a compound, the latter following the first roman letter, e.g. butyl, ethyl, isobutyl, isoquinolyl, quinolyl, s-butyl.

The general effect of the above changes will be to bring British practice more closely into line with American practice and that of other countries.

Lectures on Fibre Structure

A short full-time course of lectures on Fibre Structure will be held at the Manchester College of Science and Technology during 22nd–26th June 1959. Lecturers will describe the composition, length, and geometrical form of fibre molecules; crystal structure; the physical fine structure of fibres; and special features of particular fibres. All types of fibres will be considered.

These Post-advanced Lectures are being organised jointly by Dr. J. W. S. Hearle and Professor R. H. Peters of the Departments of Textile Industries and Textile Chemistry respectively, in collaboration with the Manchester and District Advisory Council for Further Education. Further details can be obtained from the Registrar, College of Science and Technology, Sackville Street, Manchester 1.

Abbreviations of Titles of Russian Periodicals

The abbreviations hitherto adopted in this *Journal* for the names of several Russian journals have been based on English versions of their titles, e.g. *J. Gen. Chem. U.S.S.R.* Now that complete English translations of several of these journals are becoming available, but with different pagination from the originals, it has been decided, in order to avoid confusion, to restrict these abbreviations to the English versions, and to adopt for the original journals abbreviations based on the Russian titles. The changes envisaged are set out below.

Colloid J. U.S.S.R.	Kolloidnŷĭ zhurnal	*Kolloid. zhur.*
J. Appl. Chem. U.S.S.R.	Zhurnal prikladnoĭ khimii	*Zhur. priklad. khim.*
J. Gen. Chem. U.S.S.R.	Zhurnal obshcheĭ khimii	*Zhur. obshch. khim.*
Ukrainian Chem. J.	Ukrainskiĭ khimicheskiĭ zhurnal	*Ukrain. khim. zhur.*

Asociación Española de Químicos y Coloristas Textiles

This society was founded toward the end of 1958 under the presidency of Señor José Agell. The address of its offices is Conde Borrell 187, Barcelona, Spain.

Fundamentals of Polymerisation Processes

A residential course of lectures and practical work for the non-specialist will be held at the National College of Rubber Technology, Holloway Road, London N. 7, during 6–15th July 1959. The fees, including residence at Carleton Grange, are 22 and 30 guineas for United Kingdom and overseas students respectively. Accommodation is limited, so that early application is desirable. Further details and enrolment forms are obtainable from the College.

Gordon Research Conferences

The Gordon Research Conference on Textiles for 1959 will be held during July 13–17th at Colby Junior College, New London, New Hampshire, U.S.A. Subjects to be discussed at this informal conference (cf. J.S.D.C., **74**, 423 (1958)) include the structure and modification of wool, polymer constitution and fibre properties, the cross-linking of cotton cellulose, the chemical control of wash–wear effects, and interfacial polycondensation and spinning. Additional information and application forms (which should be returned preferably by May 13th) are obtainable from W. George Park, Director, Department of Chemistry, University of Rhode Island, Kingston, R.I., U.S.A.

COLOUR INDEX

Additions and Amendments

Number 3 April 1959

In conformity with the policy stated in the preamble to the *Colour Index* it is intended to publish in the January, April, July, and October issues of the *Journal of the Society of Dyers and Colourists* and simultaneously in the *American Dyestuff Reporter* a list of additions and amendments to the Second Edition of the *Colour Index* which have been notified to the Society or the American Association of Textile Chemists and Colorists during the previous three months.

Subscribers can obtain these lists printed on one side of the paper only. For further details apply to the General Secretary, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire, England.

VOLUME 1

Additional *Colour Index* Designations and Numbers

Page 1052

C.I. 14007 Monoazo (metallised) **C.I. Acid Yellow 120**

HUE **Yellow**
Chromolan Yellow G ... ... Chem

Page 1374

C.I. 20175 Disazo **C.I. Acid Brown 213**

Coriamine Brown B2C ... ... Pol

C.I. 34907 Trisazo **C.I. Acid Brown 214**

Coriamine Brown RT ... ... Pol

Page 1404

C.I. 65008 Anthraquinone **C.I. Acid Black 97**

HUE **Greenish Grey**
Alizarine Acid Grey GG ... ... Chem

Page 1431

C.I. 14058 Monoazo **C.I. Mordant Yellow 60**

Acid Chrome Yellow 2GN ... ... Pol

Page 1549

C.I. 18092 Monoazo **C.I. Mordant Green 52**

Acid Chrome Green G ... ... Pol

Page
1584

C.I. 20151 Disazo — **C.I. Mordant Brown 90**

Hue	**Brown**
Alizarine Chrome Brown G	**Chem**

Page
1675

Diphenylamine — **C.I. Disperse Yellow 40**

Hue	**Yellow**
Serisol Fast Yellow GGL	**YDC**

Diphenylamine — **C.I. Disperse Yellow 41**

Hue	**Yellow**
Serisol Fast Yellow YGL	**YDC**

Amendments

Page	*Colour Index Designation*			
1005	**C.I. Acid Yellow 5**	*add*	**Quinoline Yellow KT ... ...**	**ICI**
1010	**C.I. Acid Yellow 17**	*add*	**Naphthazine Light Yellow 2G ...**	**BEL**
		add	**Neran Brilliant Yellow 2G ... ...**	**ICI**
1016	**C.I. Acid Yellow 29**	*add*	**Propolan Yellow 3G, 3GX ... ...**	**ICI**
1021	**C.I. Acid Yellow 40**	*delete*	**Calcocid Milling Yellow 3G ... ...**	**CCC**
	under "The following brands are similar"	*add*	**Calcocid Milling Yellow 3G ... ...**	**CCC**
1022	**C.I. Acid Yellow 42**	*add*	**Naphthalene Leather Yellow 2G ...**	**ICI**
1027	**C.I. Acid Yellow 54**	*add*	**Chromolan Yellow ELN ... ...**	**Chem**
		add	**Ultralan Yellow G ... ...**	**ICI**
1030	**C.I. Acid Yellow 61**	*add*	**Sulphonol Fast Yellow P ... ...**	**YDC**
1035	**C.I. Acid Yellow 73**	*delete*	**Calcocid Uranine SM ... ...**	**CCC**
		add	**Fluoresceine LT ... ...**	**ICI**
		add	**Fluoresceine S (free acid) ... ...**	**ICI**
1039	**C.I. Acid Yellow 85**	*add*	**Naphthalene Leather Yellow 5G ...**	**ICI**
1044	**C.I. Acid Yellow 99**	*add*	**Chromolan Yellow GRK ... ...**	**Chem**
1056	**C.I. Acid Orange 7**	*delete*	**Calcocid Orange AD ... ...**	**CCC**
1064	**C.I. Acid Orange 24**	*delete*	**Calcocid Brown YR ... ...**	**CCC**
1066	**C.I. Acid Orange 28**	*add*	**Sulphonol Fast Orange PO ... ...**	**YDC**
1078	**C.I. Acid Orange 56**	*delete*	**Calcocid Milling Orange G ... ...**	**CCC**
1083	**C.I. Acid Orange 72**	for	**Tertroxane Orange R ... ...**	**CT**
		read	**Tertroxane Orange RN ... ...**	**CT**
1084	**C.I. Acid Orange 74**	*add*	**Chromolan Orange G ... ...**	**Chem**
1085	**C.I. Acid Orange 76**	*delete*	**Calcofast Wool Orange 2R ... ...**	**CCC**
1086	**C.I. Acid Orange 79**	*add*	**Tertracid Milling Orange SRR ...**	**CT**
1096	**C.I. Acid Red 12**	*delete*	**Calcochrome Blue F4B ... ...**	**CCC**
1097	**C.I. Acid Red 14**	*add*	**Tertrochrome Blue FB ... ...**	**CT**
1101	**C.I. Acid Red 25**	*delete*	**Calcocid Scarlet 4RN ... ...**	**CCC**
		delete	**Scarlet 4RN ... ...**	**CCC**
1102	**C.I. Acid Red 26**	*delete*	**Scarlet 2RB ... ...**	**CCC**
1103	**C.I. Acid Red 27**	*delete*	**Amaranth ... ...**	**CCC**
		delete	**Calcocid Amaranth ... ...**	**CCC**
1109	**C.I. Acid Red 40**	*delete*	**Calcocid Ceresine ... ...**	**CCC**
		delete	**Seresine B ... ...**	**CCC**
		delete	**Serisine B ... ...**	**CCC**
			(see A&A, p. 4556)	
1117	**C.I. Acid Red 57**	*add*	**Kiton Fast Red 3GP ... ...**	**CAC**
1129	**C.I. Acid Red 87**	*delete*	**D&C Red No. 22 ... ...**	**CCC**
		delete	**Eosine BP ... ...**	**CCC**
1130	**C.I. Acid Red 88**	*delete*	**Calcocid Fast Red S ... ...**	**CCC**

Page	*Colour Index Designation*		
1132	C.I. Acid Red 92	*delete*	Calcocid Phloxine BN, JN CCC
		delete	Phloxine JN CCC
1133	C.I. Acid Red 94	*delete*	Calcocid Rose Bengale CCC
1141	C.I. Acid Red 114	*add*	Midlon Red PRS ... ... Chem
1149	C.I. Acid Red 129	*add*	Carbolan Rubine 2B ICI
1151	C.I. Acid Red 134	*delete*	Calcocid Milling Red BC CCC
		add	Coomassie Bordeaux B ICI
1158	C.I. Acid Red 152	*add*	Tertracid Milling Red A5BL ... ... CT
1160	C.I. Acid Red 158	*add*	Coomassie Red BR ICI
1167	C.I. Acid Red 179	*add*	Chromolan Bordeaux R... ... ... Chem
1168	C.I. Acid Red 180	*delete*	Calcofast Wool Red RN CCC
1169	C.I. Acid Red 183	*delete*	Calcofast Wool Red GA CCC
		add	Chromolan Red GRE ... ... Chem
1171	C.I. Acid Red 186	*add*	Chromolan Pink B ... ... Chem
1181	C.I. Acid Red 214	*add*	Chromolan Red BEN ... ... Chem
1193	C.I. Acid Violet 5	*add*	Tertracid Brilliant Red 10B ... ... CT
1207	C.I. Acid Violet 34	for	Calcocid Alizarine Violet NC CCC
		read	Calcocid Alizarine Violet RNC ... CCC
1214	C.I. Acid Violet 49	*add*	Tertracid Brilliant Violet 6B ... ... CT
1217	C.I. Acid Violet 56	*add*	Chromolan Violet 3R ... ... Chem
1218	C.I. Acid Violet 58	*delete*	Calcofast Wool Violet 6R CCC
1229	C.I. Acid Blue 1	*delete*	Calcocid Blue VS CCC
1232	C.I. Acid Blue 7	*add*	Disulphine Blue AN ICI
		add	Disulphine Lake Blue AN ICI
1236	C.I. Acid Blue 15	*add*	Tertracid Milling Brilliant Blue B ... CT
1239	C.I. Acid Blue 22	*add*	Calcocid Ink Blue R CCC
		add	Ink Blue A ICI
		add	Soluble Paper Blue W ICI
1249	C.I. Acid Blue 45	*delete*	Calcocid Alizarine Blue SAPX ... CCC
1271	C.I. Acid Blue 93	*delete*	Calcocid Ink Blue R CCC
1283	C.I. Acid Blue 120	*delete*	Calcocid Navy Blue R CCC
1297	C.I. Acid Blue 154	*add*	Chromolan Blue RR ... ... Chem
1299	C.I. Acid Blue 158	*add*	Chromolan Blue GG ... ... Chem
1316	C.I. Acid Green 20	*add*	Tertracid Green Black B ... ... CT
1334	C.I. Acid Brown 14	*add*	Naphthalene Leather Brown AD ... ICI
1335	C.I. Acid Brown 17	*add*	Tertracid Milling Brown AR ... ... CT
		add	Tertrochrome Brown AR ... ... CT
1339	C.I. Acid Brown 27	*add*	Carbolan Brown BL ICI
1375	C.I. Acid Black 1	*delete*	Calcocid Blue Black 10BP CCC
1376	C.I. Acid Black 2	*add*	Nigrosine HN ICI
1384	C.I. Acid Black 24	*add*	Tertracid Fast Black 2B ... ... CT
1391	C.I. Acid Black 43	*add*	Chromolan Black RR ... ... Chem
1395	C.I. Acid Black 52	*add*	Chromolan Black WA ... ... Chem
		add	Ultralan Leather Black RN ICI
1411	C.I. Mordant Yellow 10	*add*	Tertrochrome Yellow R ... ... CT
1412	C.I. Mordant Yellow 12	*add*	Metachrome Yellow MYS ... ... BEL
1413	C.I. Mordant Yellow 14	*delete*	Calcochrome Yellow 3G CCC
1433	C.I. Mordant Orange 1	*add*	Metachrome Orange R ... ... BEL
1464	C.I. Mordant Red 27	*add*	Solochrome Brilliant Red 3B ICI
1471	C.I. Mordant Red 48	*add*	Chromazol Red G ICI
1478	C.I. Mordant Red 71	*add*	Tertrochrome Bordeaux AB ... ... CT
1481	C.I. Mordant Violet 1	*add*	Solochrome Brilliant Violet R... ... ICI
1504	C.I. Mordant Blue 3	*add*	Tertrochrome Cyanine CR ... ... CT
1520	C.I. Mordant Blue 42	*add*	Tertrochrome Blue CR ... ... CT
1527	C.I. Mordant Blue 60	*add*	Chromazol Brilliant Blue 4G ICI
1541	C.I. Mordant Green 26	*add*	Solochromate Fast Olive B ICI
1551	C.I. Mordant Brown 1	*delete*	Calcochrome Brown EBL CCC
		add	Metachrome Brown EB ... ... BEL
1552	C.I. Mordant Brown 4	*add*	Metachrome Brown BR ... ... BEL
1555	C.I. Mordant Brown 12	*add*	Metachrome Brown R ... ... BEL
1558	for C.I. 20150 Monoazo	*read*	C.I. 20150 Disazo

Page	*Colour Index Designation*			
1561	**C.I. Mordant Brown 24**	*add*	**Metachrome Brown 6G ... ... BEL**	
1565	**C.I. Mordant Brown 33**	*add*	**Tertrochrome Brown RH ... ... CT**	
1571	**C.I. Mordant Brown 48**	*add*	**Metachrome Brown B ... ... BEL**	
1578	**C.I. Mordant Brown 66**	*add*	**Metachrome Olive 2G ... ... BEL**	
1579	**C.I. Mordant Brown 70**	*delete*	**Calcochrome Brown ROC ... ... CCC**	
1585	**C.I. Mordant Black 1**	for	**Tertrochrome Black A ... ... CT**	
		read	**Tertrochrome Black GA ... ... CT**	
1588	**C.I. Mordant Black 7**	*add*	**Tertrochrome Black PBB ... ... CT**	
1591	**C.I. Mordant Black 13**	*delete*	**Calcochrome Alizarine Blue Black 3B CCC**	
1619	**C.I. Basic Yellow 1**	*delete*	**Calcozine Flavine TG ... ... CCC**	
	C.I. Basic Yellow 2	*add*	**Auramine O ... ... ICI**	
1623	**C.I. Basic Orange 2**	*add*	**Chrysoidine YN ... ... ICI**	
1625	**C.I. Basic Orange 10**	*delete*	**Calcozine Phosphine 3B ... ... CCC**	
1627	**C.I. Basic Orange 16**	*delete*	**Calcozine Phosphine 2RNC ... ... CCC**	
1629	**C.I. Basic Red 1**	*add*	**Rhodamine 6GBN ... ... ICI**	
1633	**C.I. Basic Violet 1**	*add*	**Methyl Paper Violet 2BN ... ... ICI**	
1634	**C.I. Basic Violet 3**	*add*	**Methyl Violet 10BO ... ... ICI**	
1636	**C.I. Basic Violet 10**	*add*	**Rhodamine BN ... ... ICI**	
1637	**C.I. Basic Violet 14**	*add*	**Magenta Large Crystals... ... ... ICI**	
		add	**Magenta P ... ... ICI**	
1639	**C.I. Basic Blue 1**	*add*	**Acronol Brilliant Blue 6G ... ... ICI**	
1641	**C.I. Basic Blue 7**	*add*	**Victoria Pure Blue BO ... ... ICI**	
1642	**C.I. Basic Blue 9**	*delete*	**Ext. D&C Blue No. 1 ... ... CCC**	
		add	**Methylene Blue 2BN ... ... ICI**	
1647	**C.I. Basic Green 1**	*add*	**Brilliant Green YN ... ... ICI**	
1648	**C.I. Basic Green 4**	*add*	**Malachite Green CB ... ... ICI**	
1650	**C.I. Basic Brown 4**	*add*	**Bismarck Brown FR ... ... ICI**	
1659	**C.I. Disperse Yellow 1**	*add*	**Dispersol Printing Yellow A ... ... ICI**	
1660	**C.I. Disperse Yellow 3**	*add*	**Serinyl Hosiery Yellow GD ... ... YDC**	
1669	**C.I. Disperse Yellow 27**	*add*	**Serisol Fast Yellow GWD ... ... YDC**	
1672	**C.I. Disperse Yellow 34**	*add*	**Serisol Fast Yellow 4RGL ... ... YDC**	
1677	**C.I. Disperse Orange 1**	*add*	**Dispersol Printing Orange A ... ... ICI**	
1678	**C.I. Disperse Orange 3**	*delete*	**Calcosyn Orange 2RN ... ... CCC**	
		add	**Serilan Orange GXD ... ... YDC**	
1683	**C.I. Disperse Orange 13**	*add*	**Serilene Orange BL ... ... YDC**	
1687	**C.I. Disperse Red 1**	*add*	**Serinyl Hosiery Scarlet BD ... ... YDC**	
1689	**C.I. Disperse Red 5**	*add*	**Serisol Fast Rubine BD... ... ... YDC**	
1692	**C.I. Disperse Red 11**	*add*	**Duranol Printing Red X3B ... ... ICI**	
1696	**C.I. Disperse Red 19**	*add*	**Serinyl Hosiery Scarlet GD ... ... YDC**	
1697	**C.I. Disperse Red 22**	*add*	**Serisol Fast Pink RGL ... ... YDC**	
1700	**C.I. Disperse Red 31**	*delete*	**Calcosyn Scarlet GC ... ... CCC**	
1702	**C.I. Disperse Red 35**	*add*	**Serisol Fast Red NGGL ... ... YDC**	
1705	**C.I. Disperse Violet 1**	*delete*	**Calcosyn Red Violet R ... ... CCC**	
1706	**C.I. Disperse Violet 4**	*delete*	**Calcosyn Violet B ... ... CCC**	
1713	**C.I. Disperse Blue 1**	*delete*	**Calcosyn Blue 2BG ... ... CCC**	
		add	**Serinyl Blue 3G ... ... YDC**	
1714	**C.I. Disperse Blue 3**	*add*	**Duranol Printing Blue B ... ... ICI**	
		add	**Serinyl Hosiery Blue BG ... ... YDC**	
		add	**Serisol Brilliant Blue BP ... ... YDC**	
		delete	**Calcosyn Sapphire Blue 2GS ... ... CCC**	
		delete	**Calcosyn Sapphire Blue R ... ... CCC**	
	under "The following brands are similar"	*add*	**Calcosyn Sapphire Blue 2GS ... ... CCC**	
		add	**Calcosyn Sapphire Blue R ... ... CCC**	
1716	**C.I. Disperse Blue 7**	*add*	**Duranol Printing Blue Green B ... ICI**	
1718	**C.I. Disperse Blue 11**	*add*	**Serisol Diazo Navy Blue RD ... ... YDC**	
1725	**C.I. Disperse Blue 26**	*add*	**Duranol Printing Blue G ... ... ICI**	
		add	**Serilene Blue GL, 2GL... ... ... YDC**	
1727	**C.I. Disperse Blue 33** under "The following brand is similar"	*delete*	**Calcosyn Discharge Blue BN ... ... CCC**	
1728	**C.I. Disperse Blue 35**	*delete*	**Serisol Dark Blue B ... ... YDC**	
		add	**Duranol Dark Blue T ... ... ICI**	
		add	**Serilene Dark Blue G ... ... YDC**	

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1735	**C.I. Disperse Black 2**	*add*	**Dispersol Diazo Black 2BN ... ... ICI**
1738	**C.I. Disperse Black 9**	*delete*	**Calcosyn Diazo Black GHS ... ... CCC**
		add	**Serisol Diazo Black 3G ... ... YDC**
1791	**C.I. Food Blue 3**	*add*	**Edicol Supra Blue VR ... ... ICI**

VOLUME 2

Additional *Colour Index* Designations and Numbers

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C.I. 29232 Disazo — **C.I. Direct Violet 87**

Helion Violet 3BL ... ... Pol

C.I. 22046 Disazo — **C.I. Direct Violet 88**

Cresotine Dark Violet ... ... Pol

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C.I. 31951 Trisazo — **C.I. Direct Blue 183**

Diazo Blue 2B ... ... Pol

C.I. 29128 Disazo — **C.I. Direct Blue 184**

Helion Blue 3RL ... ... Pol

C.I. 29130 Disazo — **C.I. Direct Blue 185**

Helion Navy Blue GL ... ... Pol

C.I. 34139 Trisazo — **C.I. Direct Blue 186**

HUE **Reddish Navy**
Saturn Blue LRR ... ... Chem

C.I. 34146 Trisazo — **C.I. Direct Blue 187**

Helion Blue RL ... ... Pol

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C.I. 35415 Polyazo — **C.I. Direct Black 100**

Cresotine Grey B... ... ... Pol

C.I. 34100 Trisazo — **C.I. Direct Black 101**

Helion Grey BL ... ... Pol

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C.I. 53218 **C.I. Sulphur Brown 62**

HUE **Brownish Olive → Yellowish Brown**
Sulfogen Black Brown 2N ... ... Chem

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C.I. 59856 Anthraquinone **C.I. Vat Blue 51**

Helanthrene Navy Blue LK ... ... Pol

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C.I. 73685 Indigoid **C.I. Vat Green 25**

HUE **Green**
Cibanone Green GC ... ... CAC, Ciba

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C.I. Azoic Blue 27

HUE **Greenish Navy**
Citazene Blue CR ... ... Acna

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C.I. ▼Azoic Black 8

For Polyester fibre

HUE **Greenish Black**
Serilene Azoic Black B2 ... ... YDC

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C.I. 12470 Monoazo **C.I. Pigment Orange 22**

HUE **Reddish Orange**
Versal Orange GR ... ... Chem

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C.I. Solvent Yellow 52

HUE **Reddish Yellow**
Seriplas Yellow A ... ... YDC

Page 2842

C.I. Solvent Orange 35

HUE **Bright Orange**
Seriplas Orange YL ... ... YDC

Amendments

Page	*Colour Index Designation*		
2005	**C.I. Direct Yellow 6**	*add*	**Chlorazol Paper Yellow FG ... ... ICI**
2007	**C.I. Direct Yellow 9**	*add*	**Chlorazol Yellow DP ... ... ICI**
2008	**C.I. Direct Yellow 11**	*add*	**Chlorazol Paper Yellow R ... ... ICI**
2009	**C.I. Direct Yellow 12**	*delete*	**Chrysophenine ESN ... ... CCC**
		add	**Tertrodirect Yellow CG ... ... CT**
2015	**C.I. Direct Yellow 28**	*add*	**Tertrodirect Fast Yellow FRL ... ... CT**
2016	**C.I. Direct Yellow 29**	*add*	**Tertrodirect Fast Yellow RT ... ... CT**
		delete	**C.I. Direct Yellow 30**
2034	**C.I. Direct Yellow 72**	*add*	**Icin Soga Koening RN ... ... ICI**
2037	**C.I. Direct Orange 1**	*add*	**Tertrodirect Orange PG ... ... CT**
2044	**C.I. Direct Orange 18**	*add*	**Chlorazol Orange Brown RNP ... ICI**
2052	**C.I. Direct Orange 40**	*delete*	**Tertrodirect Light Brown 2R ... ... CT**
		add	**Diazine Fast Brown YRL ... ... BEL**
		add	**Durazol Orange Brown GP ... ... ICI**
2063	**C.I. Direct Orange 73**	*delete*	**Calcoform Yellow RXD ... ... CCC**
		add	**Chlorazol Diazo Orange 2G ... ... ICI**
2079	**C.I. Direct Red 10**	*delete*	**Calcomine Garnet B ... ... CCC**
2080	**C.I. Direct Red 13**	*delete*	**Calcomine Bordeaux 6BN ... ... CCC**
2081	**C.I. Direct Red 16**	*delete*	**Pheno Red 6BX ... ... CCC**
2082	**C.I. Direct Red 20**	*delete*	**Calcomine Pink RX ... ... CCC**
		delete	**Pheno Pink RX ... ... CCC**
2083	**C.I. Direct Red 23**	*add*	**Chlorazol Fast Scarlet 4BSP ... ... ICI**
2087	**C.I. Direct Red 31**	for	**Tertrodirect Pink RD ... ... CT**
		read	**Tertrodirect Pink DR ... ... CT**
		delete	**Calcomine Red 12B ... ... CCC**
2092	**C.I. Direct Red 45**	*delete*	**Calcomine Brilliant Pink SX ... ... CCC**
2097	**C.I. Direct Red 61**	*add*	**Tertrodirect Brilliant Purpurine 10B CT**
2103	**C.I. Direct Red 80**	*add*	**Tertrodirect Fast Red F3B ... ... CT**
2105	**C.I. Direct Red 84**	*add*	**Tertrodirect Fast Brown 3RL ... ... CT**
2108	**C.I. Direct Red 94**	*delete*	**Calcodur Brown 4RL ... ... CCC**
2110	**C.I. Direct Red 99**	for	**Disazo** (metallised)
		read	**C.I. 29167** Disazo (metallised)
		add	**Helion Bordeaux BL ... ... Pol**
		add	**Saturn Bordeaux LB ... ... Chem**
2114	**C.I. Direct Red 111**	*add*	**Tertrodirect Fast Brown 4RD ... CT**
2116	**C.I. Direct Red 116**	*add*	**Tertrodirect Fast Brown 2R ... ... CT**
2118	**C.I. Direct Red 121**	*add*	**Tertrodiazo Bordeaux FBL ... ... CT**
2119	**C.I. Direct Red 123**	*add*	**Tertrodiazo Brilliant Scarlet ROA ... CT**
2130	**C.I. Direct Red 154**	*add*	**Tertrodiazo Red 8BL ... ... CT**
2145	**C.I. Direct Violet 7**	*add*	**Chlorazol Fast Helio 2RKP ... ... ICI**
2148	**C.I. Direct Violet 14**	*delete*	**Calcomine Brilliant Rubine B ... ... CCC**
2150	**C.I. Direct Violet 22**	*delete*	**Calcomine Violet B ... ... CCC**
		delete	**Pheno Violet B ... ... CCC**
2156	**C.I. Direct Violet 46**	for	**Monoazo** (metallised)
		read	**C.I. 17515** Monoazo (metallised)
		delete	**Oxanal Violet RLL ... ... Ciba**
		add	**Direct Supra Light Violet MRL ... ICS**
		add	**Saturn Violet LRL ... ... Chem**
			See the instructions for C.I. Direct Violet 55
2159		*delete*	**C.I. Direct Violet 55**
			Transfer the dye and information to C.I. Direct Violet 46
2163	**C.I. Direct Violet 67**	*delete*	**Calcoform Violet BD ... ... CCC**
2170	**C.I. Direct Blue 2**	*delete*	**Calcomine Diazo Black BHGD ... CCC**
		delete	**Pheno Navy Blue B ... ... CCC**
2179	**C.I. Direct Blue 22**	*delete*	**Calcomine Blue RW ... ... CCC**
2181	**C.I. Direct Blue 26**	for	**Calcomine Dark Blue B ... ... CCC**
		read	**Calcomine Dark Blue BN ... ... CCC**
2193	**C.I. Direct Blue 67**	*delete*	**Calcodur Blue RL ... ... CCC**
2196	**C.I. Direct Blue 74**	*add*	**Durazol Burl Blue 3R ... ... ICI**
2198	**C.I. Direct Blue 78**	*add*	**Tertrodirect Fast Blue SG ... ... CT**
2200	**C.I. Direct Blue 84**	*add*	**Tertrodirect Fast Blue SGL ... ... CT**
2201	**C.I. Direct Blue 86**	*add*	**Tertrodirect Fast Turquoise GL ... CT**

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2201	**C.I. Direct Blue 87**	*add*	**Durazol Blue 6GW ... ... ICI**	
		add	**Durazol Printing Blue 6G ... ... ICI**	
2204	**C.I. Direct Blue 96**	*add*	**Tertrodirect Fast Blue 7GL ... ... CT**	
2208	**C.I. Direct Blue 106**	*add*	**Durazol Sky Blue G ... ... ICI**	
		add	**Durazol Sky Blue GP ... ... ICI**	
2209	**C.I. Direct Blue 109**	*add*	**Durazol Brilliant Blue B ... ... ICI**	
		add	**Durazol Brilliant Blue BP ... ... ICI**	
2213	**C.I. Direct Blue 120**	*add*	**Tertrodiazo Indigo Blue BR ... ... CT**	
	C.I. Direct Blue 120 or 120A	*delete*	**Calcomine Diazo Blue BD ... ... CCC**	
	C.I. Direct Blue 121	*add*	**Tertrodiazo Indigo Blue RM ... ... CT**	
2215	**C.I. Direct Blue 126**	*add*	**Tertrodiazo Blue 2R ... ... CT**	
2218	**C.I. Direct Blue 135**	*add*	**Tertrodiazo Indigo Blue 3RL ... ... CT**	
2223	**C.I. Direct Blue 148**	*add*	**Tertrodirect Fast Copper Blue GL ... CT**	
2239	**C.I. Direct Green 6**	*add*	**Chlorazol Paper Green BN ... ... ICI**	
2240	**C.I. Direct Green 8**	*delete*	**Calcomine Green SL ... ... CCC**	
2241	**C.I. Direct Green 12**	*delete*	**Calcomine Green BT ... ... CCC**	
2247	**C.I. Direct Green 26**	*add*	**Durazol Green BL ... ... ICI**	
	C.I. Direct Green 27	*add*	**Durazol Green 5B ... ... ICI**	
2250	**C.I. Direct Green 34**	*add*	**Durazol Green D... ... ... ICI**	
2256	**C.I. Direct Green 47**	*delete*	**Calcoform Green BLD ... ... CCC**	
2264	**C.I. Direct Brown 2**	*add*	**Chlorazol Brown MP ... ... ICI**	
2272	**C.I. Direct Brown 25**	*delete*	**Calcomine Catechu GN... ... ... CCC**	
2273	**C.I. Direct Brown 27**	for	**Tertrodirect Brown CBO ... ... CT**	
		read	**Tertrodirect Brown CB ... ... CT**	
	C.I. Direct Brown 28	*delete*	**Calcomine Chrome Brown BS ... CCC**	
2275	**C.I. Direct Brown 31**	*add*	**Direct Brown FS ... ... Chem**	
2276	**C.I. Direct Brown 33**	*delete*	**Calcomine Catechu LB ... ... CCC**	
2283	**C.I. Direct Brown 52**	*add*	**Tertrodirect Bronze G ... ... CT**	
2292	**C.I. Direct Brown 79**	*add*	**Direct Brown 3GN ... ... Chem**	
2295	**C.I. Direct Brown 95**	*add*	**Diazine Fast Brown RSL ... ... BEL**	
	under "The following brands are similar"	*add*	**Diazine Fast Brown GL ... ... BEL**	
2313	**C.I. Direct Brown 153**	*add*	**Azogen Brown R ... ... Chem**	
2326	**C.I. Direct Black 4**	*delete*	**Calcomine Black R ... ... CCC**	
2332	**C.I. Direct Black 19**	*add*	**Tertrodirect Black V ... ... CT**	
2335	**C.I. Direct Black 28**	*delete*	**Tertrodirect Black V ... ... CT**	
2338	**C.I. Direct Black 38**	*delete*	**Calcomine Black G ... ... CCC**	
		add	**Calcomine Black EXL ... ... CCC**	
2339		*add*	**Chlorazol Leather Black ENP ... ICI**	
2349	**C.I. Direct Black 71**	*add*	**Tertrodirect Fast Grey VGL ... ... CT**	
2353	**C.I. Direct Black 80**	*add*	**Tertrodiazo Black OB ... ... CT**	
2363	**C.I. Sulphur Yellow 2**			
	under "Applied by method 1"	*add*	**Sulfogen Yellow R ... ... Chem**	
		add	**Tertrosulphur Yellow 2G ... ... CT**	
2364	**C.I. Sulphur Yellow 4**			
	under "Applied by method 1"	*add*	**Tertrosulphur Yellow 6G ... ... CT**	
2366	**C.I. Sulphur Yellow 9**	*add*	**Sulfogen Yellow G ... ... Chem**	
2369	**C.I. Sulphur Orange 1**			
	under "Applied by method 1"	*add*	**Sulfogen Brown RS ... ... Chem**	
		add	**Tertrosulphur Orange CN ... ... CT**	
2370	**C.I. Sulphur Orange 3**			
	under "Applied by method 1"	*add*	**Sulfogen Orange RK ... ... Chem**	
		add	**Tertrosulphur Orange TR ... ... CT**	
2373	**C.I. Sulphur Red 6**			
	under "Applied by method 1"	*delete*	**Calcogene Violet Brown CF Conc. ... CCC**	
		add	**Sulfogen Red Brown BK ... ... Chem**	
2376	**C.I. Sulphur Violet 4**			
	under "Applied by method 1"	*delete*	**Calcogene Claret G ... ... CCC**	
		add	**Sulfogen Purple C ... ... Chem**	
2378	**C.I. Sulphur Blue 3**	*add*	**Sulfogen Blue 6B ... ... Chem**	
2379	**C.I. Sulphur Blue 5**			
	under "Applied by method 1"	*add*	**Tertrosulphur Direct Blue B, VBR ... CT**	
2381	**C.I. Sulphur Blue 10**			
	under "Applied by method 1"	*add*	**Sulfogen Blue FBL ... ... Chem**	
2382	**C.I. Sulphur Blue 11**			
	under "Applied by method 1"	*add*	**Sulfogen Blue 2RL ... ... Chem**	
		add	**Tertrosulphur Direct Blue RL ... CT**	

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2384	**C.I. Sulphur Blue 15**			
	under "Applied by method 1"	*add*	**Sulfogen Green B ... ... Chem**	
		add	**Sulfogen Green Blue CV ... ... Chem**	
2385	**C.I. Sulphur Green 2**			
	under "Applied by method 1"	*delete*	**Calcogene Green YS-CF ... ... CCC**	
		add	**Tertrosulphur Green B ... ... CT**	
2386	**C.I. Sulphur Green 3**			
	under "Applied by method 1"	*add*	**Sulfogen Green 3G ... ... Chem**	
2396	**C.I. Sulphur Brown 10**			
	under "Applied by method 1"	*delete*	**Calcogene Tan Y-CF extra conc. ... CCC**	
		add	**Tertrosulphur Cutch MG ... ... CT**	
2397	**C.I. Sulphur Brown 12**			
	under "Applied by method 1"	*add*	**Tertrosulphur Bordeaux B ... ... CT**	
2398	**C.I. Sulphur Brown 14**			
	under "Applied by method 1"	*delete*	**Calcogene Dark Brown CN-CF ... CCC**	
2413	**C.I. Sulphur Black 1**			
	under "Applied by method 1"	*add*	**Sulfogen Black BWLE ... ... Chem**	
2427	**C.I. Vat Yellow 1**	*delete*	**Calcosol Yellow G ... ... CCC**	
2439	**C.I. Vat Yellow 26**	*add*	**Caledon Printing Yellow 5GKN ... ICI**	
2447	**C.I. Vat Orange 5**	*delete*	**Calcoloid Orange RD ... ... CCC**	
		add	**Durindone Orange RP ... ... ICI**	
2452	**C.I. Vat Orange 15**	for	**Calcosol Golden Orange 3G ... ... CCC**	
		read	**Calcoloid Golden Orange 3G ... ... CCC**	
2461	**C.I. Vat Red 11**	*add*	**Durindone Pink 2BP ... ... ICI**	
2462	**C.I. Vat Red 13**	*delete*	**Calcosol Red 2B ... ... CCC**	
2465	**C.I. Vat Red 21**	*add*	**Caledon Red 4B ... ... ICI**	
2469	**C.I. Vat Red 31**	*add*	**Caledon Brilliant Red 5B ... ... ICI**	
2471	**C.I. Vat Red 37**	*add*	**Caledon Printing Scarlet G ... ... ICI**	
		add	**Caledon Scarlet G ... ... ICI**	
2473	**C.I. Vat Red 41**	*add*	**Durindone Red BP ... ... ICI**	
2475	**C.I. Vat Red 45**	*add*	**Durindone Scarlet YP ... ... ICI**	
2479	**C.I. Vat Violet 1**	*add*	**Calcoloid Violet 4RD ... ... CCC**	
2480	**C.I. Vat Violet 2**	*delete*	**Calcosol Violet 6RD ... ... CCC**	
		add	**Calcoloid Violet 6RD ... ... CCC**	
		add	**Durindone Red 3BP ... ... ICI**	
2481	**C.I. Vat Violet 3**	*add*	**Durindone Magenta BP ... ... ICI**	
2485	**C.I. Vat Violet 9**	*add*	**Caledon Printing Violet 3B ... ... ICI**	
2488	**C.I. Vat Violet 15**	*add*	**Caledon Brilliant Violet 2B ... ... ICI**	
2491	**C.I. Vat Blue 1**	*add*	**Indigo ND ... ... ICI**	
		add	**Vulcafor Blue A ... ... ICI**	
		add	**Vynamon Blue A ... ... ICI**	
2494	**C.I. Vat Blue 4**	for	**Calcosol Blue RS ... ... CCC**	
		read	**Calcoloid Blue RS ... ... CCC**	
2495	**C.I. Vat Blue 5**	*delete*	**Calcosol Blue 2BDG ... ... CCC**	
		add	**Durindone Blue 4BCP ... ... ICI**	
2496	**C.I. Vat Blue 6**	*delete*	**Calcoloid Blue BLN, BLNS ... ... CCC**	
		for	**Calcosol Blue BLD ... ... CCC**	
		read	**Calcoloid Blue BLD ... ... CCC**	
		add	**Calcoloid Blue BLC ... ... CCC**	
2496	**C.I. Solubilised Vat Blue 6**	*add*	**Soledon Blue 2RCX ... ... ICI**	
2497	**C.I. Vat Blue 7**	*add*	**Caledon Printing Green 6B ... ... ICI**	
2501	**C.I. Vat Blue 14**	*delete*	**Calcosol Blue GCD, GCP ... ... CCC**	
		add	**Calcoloid Blue GCD ... ... CCC**	
2503	**C.I. Vat Blue 18**	*add*	**Caledon Navy Blue ART ... ... ICI**	
2504	**C.I. Vat Blue 20**	*delete*	**Monolite Blue 3R ... ... ICI**	
		add	**Calcoloid Dark Blue BRD ... ... CCC**	
2510	**C.I. Vat Blue 35**	*delete*	**Calcosol Blue RP ... ... CCC**	
2521	**C.I. Vat Green 3**	for	**Cibanone Olive 2B℗ ... ... CAC, Ciba**	
		read	**Cibanone Olive FB ... ... CAC, Ciba**	
2522	**C.I. Vat Green 4**	*add*	**Caledon Jade Green BW ... ... ICI**	
2524	**C.I. Vat Green 8**	*delete*	**Calcosol Khaki G ... ... CCC**	
2525	**C.I. Vat Green 9**	*delete*	**Calcosol Black BB ... ... CCC**	
2531	**C.I. Vat Brown 1**	*delete*	**Calcosol Brown BR ... ... CCC**	
2532	**C.I. Vat Brown 3**	*delete*	**Calcosol Brown R, RN ... ... CCC**	
		add	**Calcoloid Brown RNB ... ... CCC**	
2533	**C.I. Vat Brown 5**	for	**Calcoloid Brown RRP ... ... CCC**	
		read	**Calcoloid Brown RRWP ... ... CCC**	
		add	**Durindone Brown GP ... ... ICI**	

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2551	**C.I. Vat Black 1**	*add*	**Durindone Printing Black BL ... ICI**
2557	**C.I. Vat Black 16**	*add*	**Helanthrene Grey 3B ... ... Pol**
2559	**C.I. Vat Black 22**	*delete*	**Calcosol Grey 2G, 2GP ... ... CCC**
		add	**Calcoloid Grey 2GN, 2GP, 2GC ... CCC**
2560	**C.I. Vat Black 25**	*delete*	**Calcosol Olive T, TCC, TRR ... ... CCC**
		add	**Calcoloid Olive TCC, TRRC ... ... CCC**
	C.I. Solubilised Vat Black 25	*add*	**Soledon Olive D ... ... ICI**
2561	**C.I. Vat Black 27**	*delete*	**Calcosol Olive R ... ... CCC**
		add	**Caledon Olive RP ... ... ICI**
2572	**C.I. Azoic Diazo Component 2**	*add*	**Amarthol Fast Orange GC Base ... Amar**
2573	**C.I. Azoic Diazo Component 3**	*add*	**Amarthol Fast Scarlet GG Base, GGS Base ... ... Amar**
2574	**C.I. Azoic Diazo Component 4**	*add*	**Amarthol Fast Garnet GBC Base ... Amar**
2578	**C.I. Azoic Diazo Component 8**	*add*	**Amarthol Fast Red GL Base ... ... Amar**
2579	**C.I. Azoic Diazo Component 9** under "Stabilised Diazo Salt"	for *read*	**Azofix Red 3GK ... ... FDC** **Azofix Red 3GL Salt ... ... FDC**
2580	**C.I. Azoic Diazo Component 10**	*add*	**Amarthol Fast Red RC Base ... ... Amar**
2586	**C.I. Azoic Diazo Component 20**	*add*	**Naphtoelan Blue BB Base ... ... Pol**
2592	**C.I. Azoic Diazo Component 32**	*add*	**Amarthol Fast Red KB Base ... ... Amar**
2593	**C.I. Azoic Diazo Component 33**	*add*	**Brentamine Fast Red FR Base ... ICI**
		add	**Brentamine Fast Red FR Salt ... ICI**
2604	**C.I. Azoic Diazo Component 44**	*add*	**Amarthol Fast Yellow GC Base ... Amar**
2610	**C.I. Azoic Diazo Component 50**	*add*	**Brentamine Fast Scarlet AT Salt ... ICI**
2616	**C.I. Azoic Coupling Component 12**	for *read*	**Brenthol ITR ... ... ICI** **Brenthol LTR ... ... ICI**
2617	**C.I. Azoic Coupling Component 13**	*add*	**Amarthol AS-SG ... ... Amar**
	C.I. Azoic Coupling Component 14	*add*	**Amarthol AS-PH ... ... Amar**
2618	**C.I. Azoic Coupling Component 18**	*add*	**Amarthol AS-D ... ... Amar**
		add	**Brentosyn OT ... ... ICI**
2620	**C.I. Azoic Coupling Component 24**	*add*	**Brenthol MA ... ... ICI**
	C.I. Azoic Coupling Component 25	*add*	**Brenthol RB ... ... ICI**
2635	**C.I. Azoic Orange 2**	*add*	**Brentogen Orange G ... ... ICI**
2645	**C.I. Azoic Red 16**	*add*	**Brentogen Bordeaux R ... ... ICI**
2657	**C.I. Azoic Violet 1**	*add*	**Brentogen Violet B ... ... ICI**
	C.I. Azoic Violet 2	*add*	**Brentogen Red Violet 2R ... ... ICI**
2662	**C.I. Azoic Blue 6**	*add*	**Brentogen Blue B ... ... ICI**
2677	**C.I. Azoic Black 3**	*delete*	**Rapidogen Black ITA ... ... G**
2684	**C.I. Oxidation Base 3**	*add*	**Soloxan Black S ... ... ICI**
		add	**Soloxan Black SN ... ... ICI**
2703	**C.I. Pigment Yellow 2**	*add*	**Tertropigment Yellow GR ... ... CT**
2707	**C.I. Pigment Yellow 12**	*add*	**Monolite Yellow GTN ... ... ICI**
	C.I. Pigment Yellow 13	*add*	**Tertropigment Fast Yellow VGR ... CT**
		add	**Tertropigment Yellow PGR ... ... CT**
2708	**C.I. Pigment Yellow 14**	*add*	**Tertropigment Fast Yellow VG ... CT**
		add	**Tertropigment Yellow BG ... ... CT**
		add	**Vynamon Yellow 2EG ... ... ICI**
2722	**C.I. Pigment Orange 13**	*add*	**Monolite Fast Paper Orange G ... ICI**
2725	**C.I. Pigment Red 2**	*add*	**Monolite Fast Paper Red 2R ... ... ICI**
2727	**C.I. Pigment Red 5**	*add*	**Monolite Fast Carmine B, BV ... ICI**
2738	**C.I. Pigment Red 44**	*add*	**Vulcafor Fast Red M ... ... ICI**
2739	**C.I. Pigment Red 48**	*add*	*Calcium salt* **Rubine Toner 2BO ... ... ICI**
2740	**C.I. Pigment Red 49**	under	*Sodium salt*
		add	**Vulcafor Red R ... ... ICI**
2748	**C.I. Pigment Red 63**	*add*	*Calcium salt* **Claret Toner 13554 ... ... ICI**
2764	**C.I. Pigment Violet 3**	*add*	**Fastel Violet R ... ... ICI**
2773	**C.I. Pigment Blue 15**	*add*	**Monastral Fast Blue BNV, BX, LBC, LBX ... ... ICI**
		add	**Monastral Fast Paper Blue B ... ... ICI**
		add	**Polymon Blue B, LB ... ... ICI**
2783	**C.I. Pigment Green 7**	*add*	**Monastral Fast Green GV, GX ... ICI**
		add	**Polymon Green G ... ... ICI**

Page	*Colour Index Designation*			
2784	**C.I. Pigment Green 10**	*add*	**Monolite Fast Green Y**	**ICI**
2785	**C.I. Pigment Green 13**	*add*	**Vulcafor Green LN**	**ICI**
2791	**C.I. Pigment Brown 1**	*add*	**Monolite Fast Brown 3GV**	**ICI**
2806	**C.I. Pigment White 18**	*add*	**Calofil A.4, B.1, E.2**	**Stu**
		add	**Calofort S, T, U**	**Stu**
		add	**Calopake F, FS, H**	**Stu**
		add	**Calopake—High Opacity**	**Stu**
		add	**Sturcal D, F, H, L, LS, M**	**Stu**
		add	**Sturcal-X**	**Stu**
2817	**C.I. Solvent Yellow 2**	*add*	**Waxoline Yellow ADM**	**ICI**
2821	**C.I. Solvent Yellow 14**	*add*	**Waxoline Yellow IM, IP**	**ICI**
2847	**C.I. Solvent Red 14**	*add*	**Seriplas Scarlet B**	**YDC**
2850	**C.I. Solvent Red 24**	*add*	**Waxoline Red OM**	**ICI**
2854	**C.I. Solvent Red 35**	*add*	**Methasol Fast Red 3B**	**ICI**
2859	**C.I. Solvent Red 53**	*add*	**Seriplas Red 2B**	**YDC**
2883	**C.I. Solvent Blue 36**	*add*	**Waxoline Blue A, AM, AP**	**ICI**
	C.I. Solvent Blue 37	*add*	**Methasol Blue RL**	**ICI**
	C.I. Solvent Blue 38	*add*	**Methasol Fast Blue 2B**	**ICI**
2927	**C.I. Developer 8**	*add*	**Beta Oxynaphthoic Acid**	**RL**
		add	**Brentosyn BB**	**ICI**
		add	**Developer BON**	**RL**
2928	**C.I. Developer 14**	*add*	**Metatoluylenediamine**	**RL**

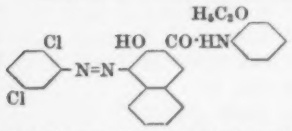
VOLUME 3

Additions and Amendments

Page *Colour Index No.*

3035 *add*

12470 **C.I. Pigment Orange 22** (*Reddish orange*) *Discoverer* — Chemapol



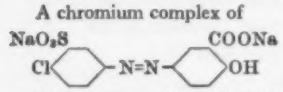
2,5-Dichloroaniline → 3-Hydroxy-2-naphth-*o*-phenetidide

3036 under **12770 C.I. Disperse Yellow 4** (*Bright yellowish green*)
add **C.I. Solvent Yellow 17** (*Greenish yellow*)

3051 *add*

14007 **C.I. Acid Yellow 120** (*Yellow*) *Discoverer* — Chemapol

A chromium complex of



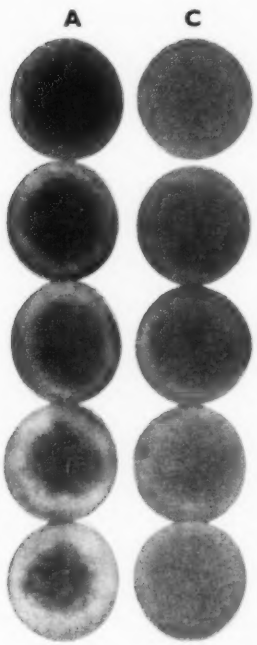
6-Chlorometanilic acid → Salicylic acid

and convert to the chromium complex

3106 for **18070**
read **18070★**

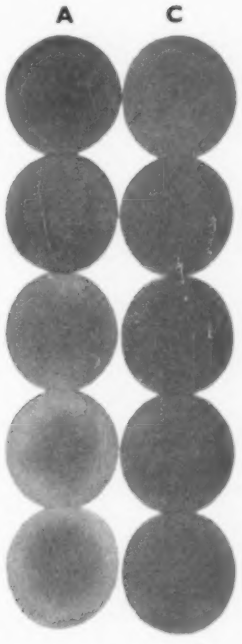
add **Calcocid Ceresine (CCC)**
add **Serisine B (CCC)**

3109 *beneath* **18736 C.I. Acid Red 180** (*Red*)
add **18736A C.I. Solvent Orange 6** (*Reddish orange*)



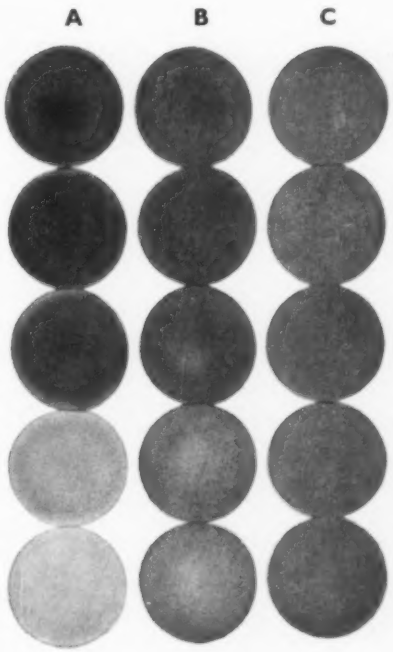
0·25% SOLAR GREY 2BL

0·25% SOLAR YELLOW 2RC



0·25% SOLAR RED 2BL

A	NO LEVELLING AGENT
B	1% LYOGEN DK
C	2% LYOGEN DK



COMBINATION SHADE

0·083% SOLAR YELLOW 2RC
0·083% SOLAR RED 2BL
0·083% SOLAR GREY 2BL

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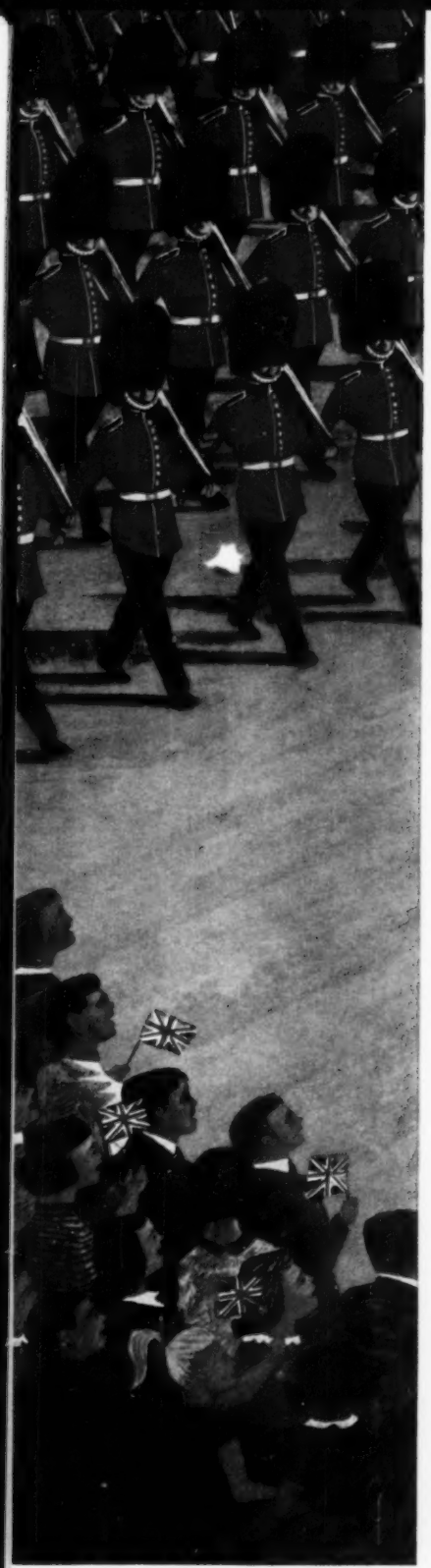

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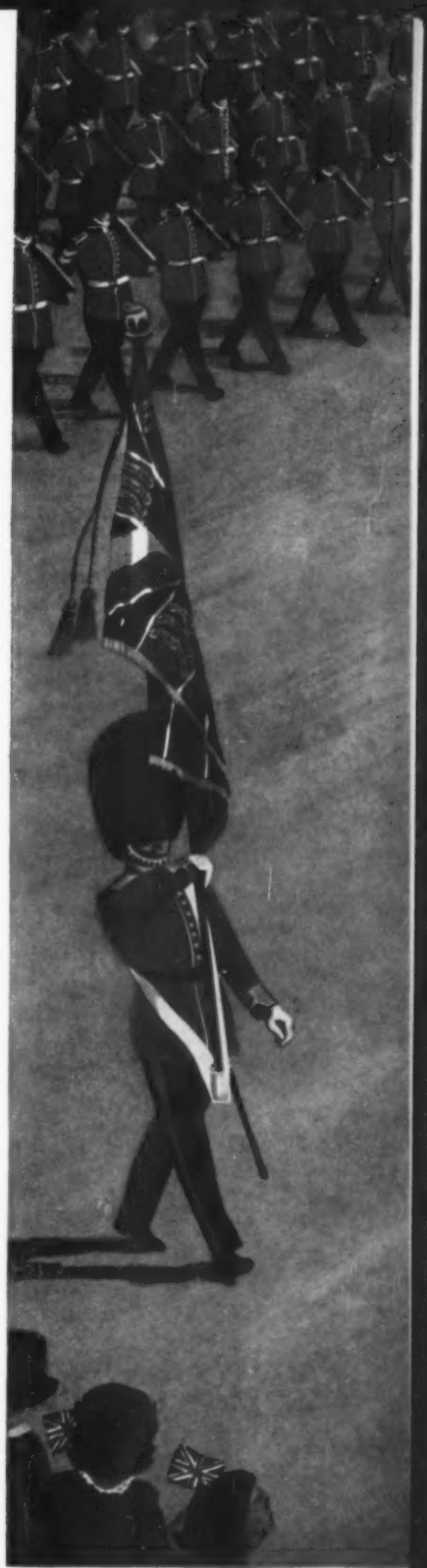

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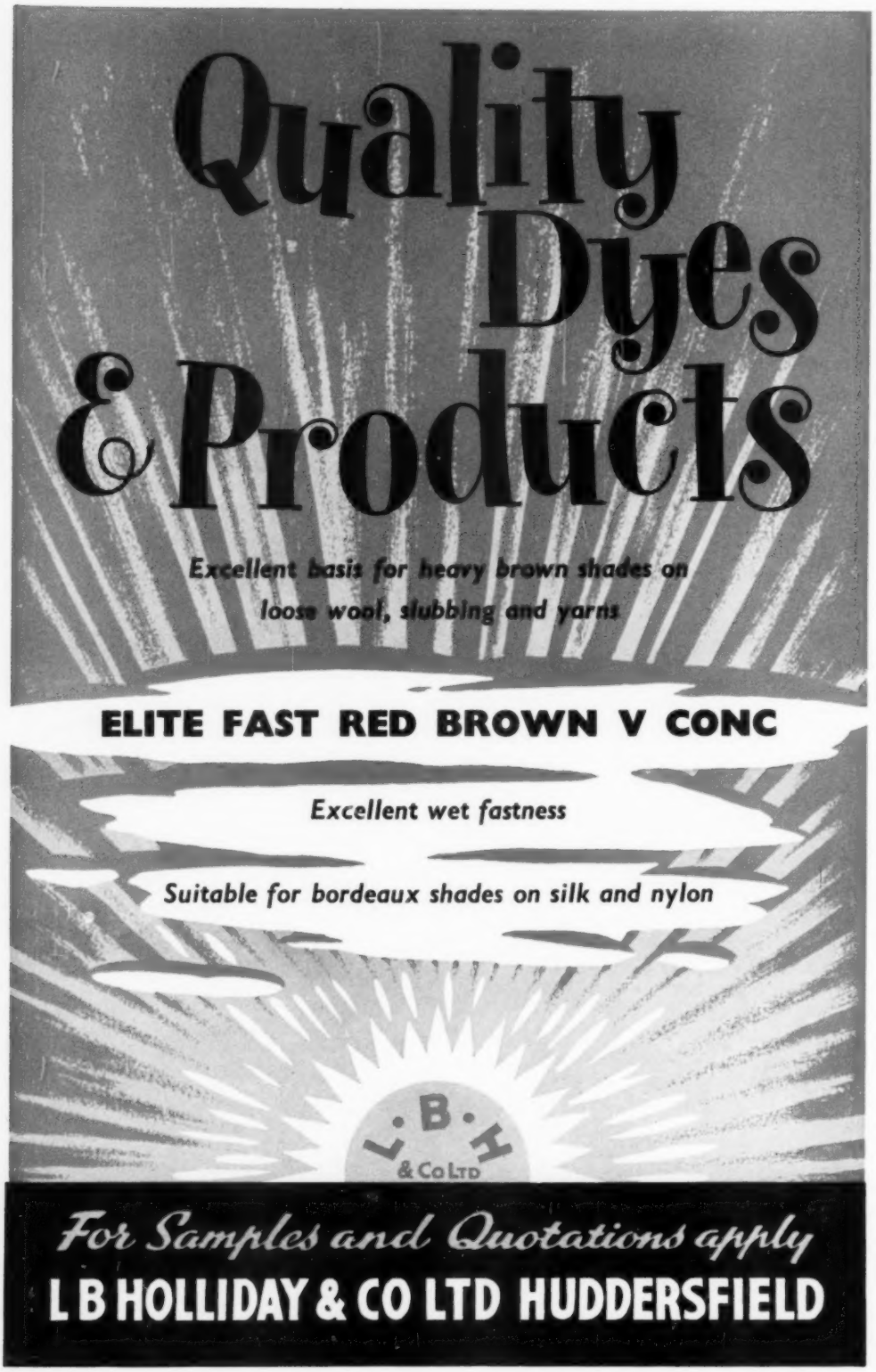
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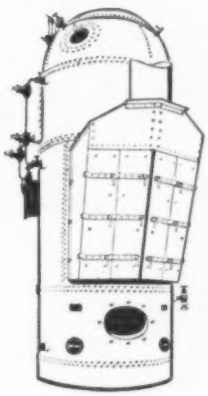

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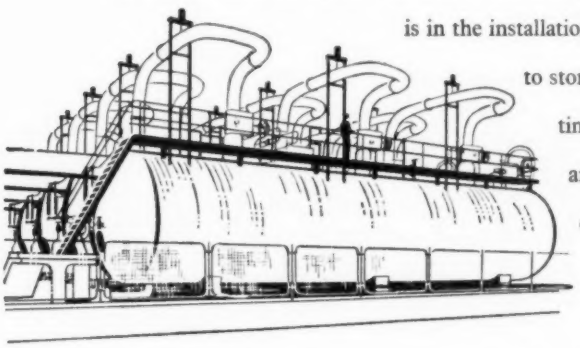

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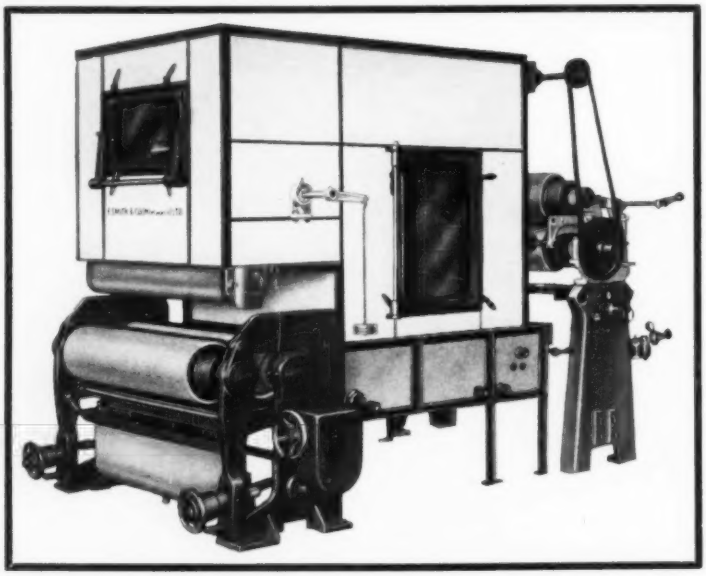

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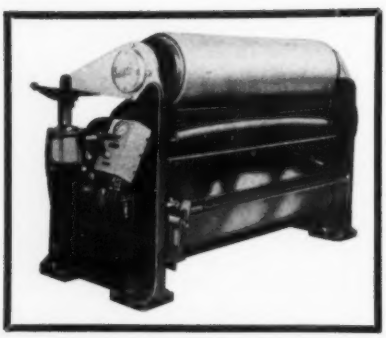

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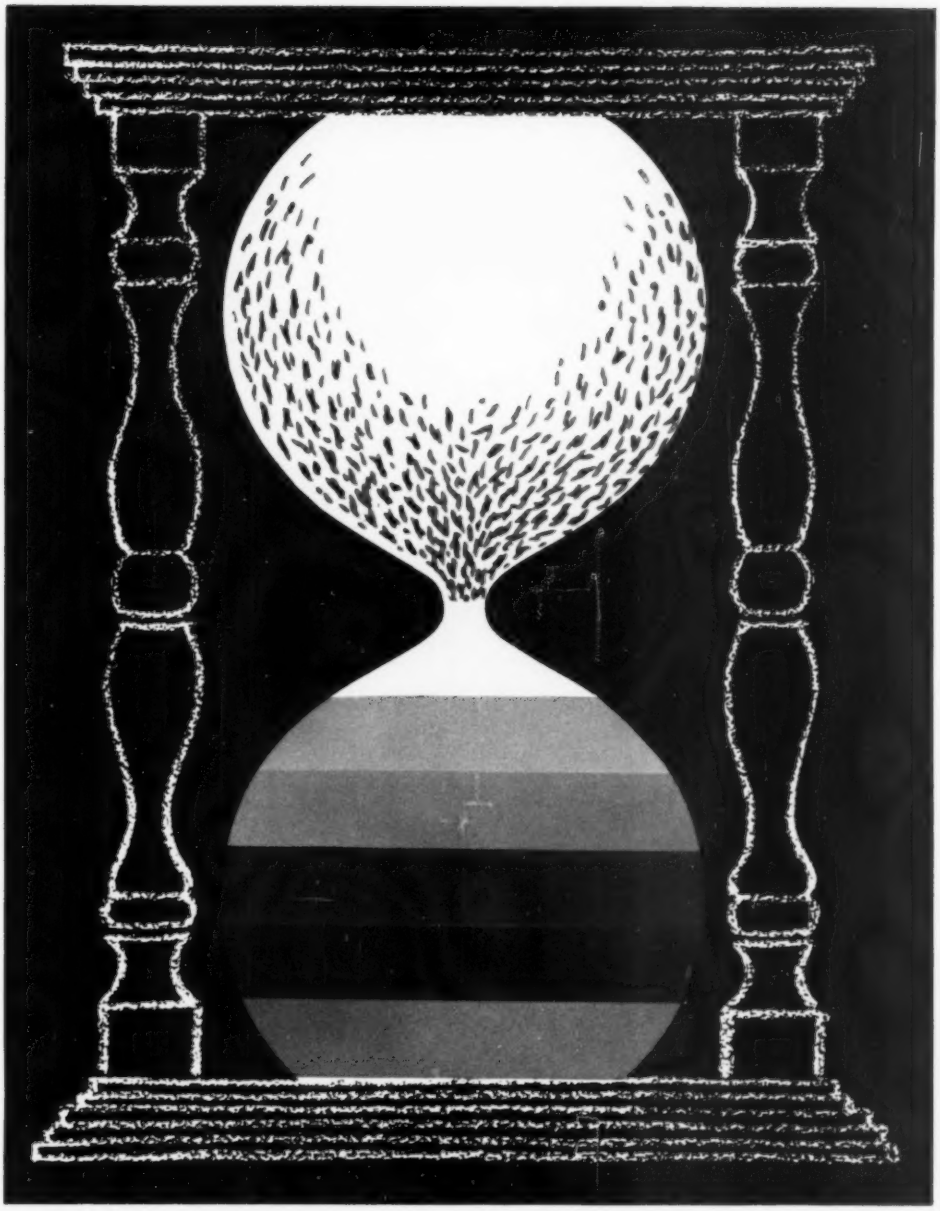

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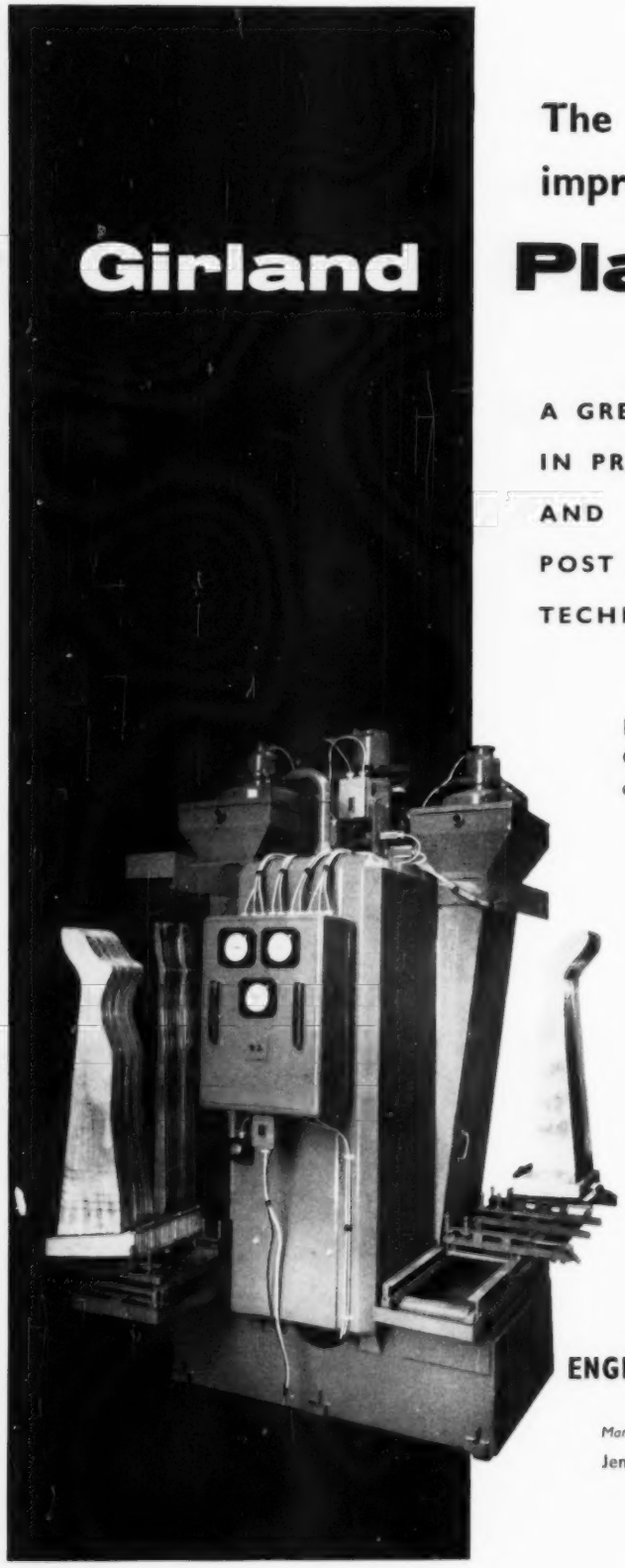

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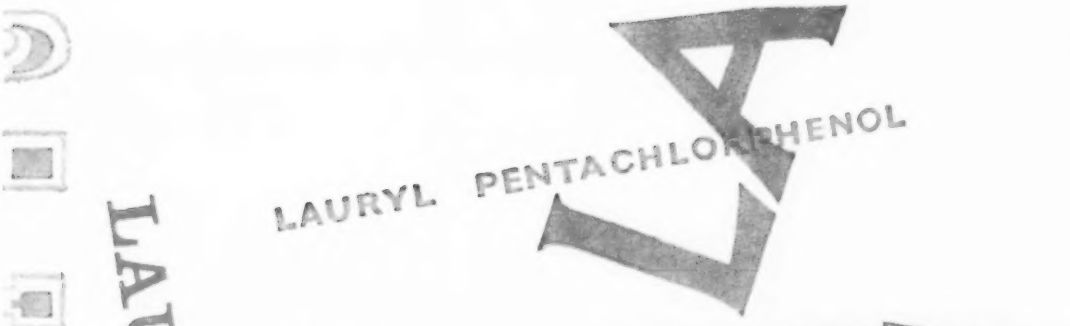

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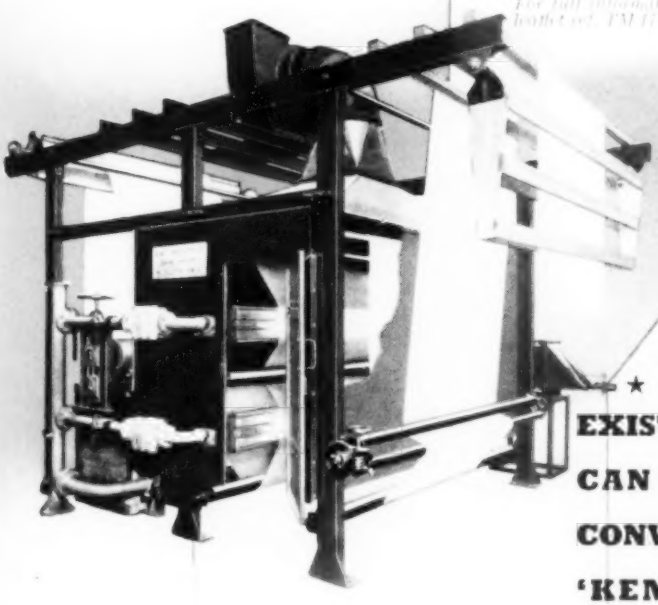

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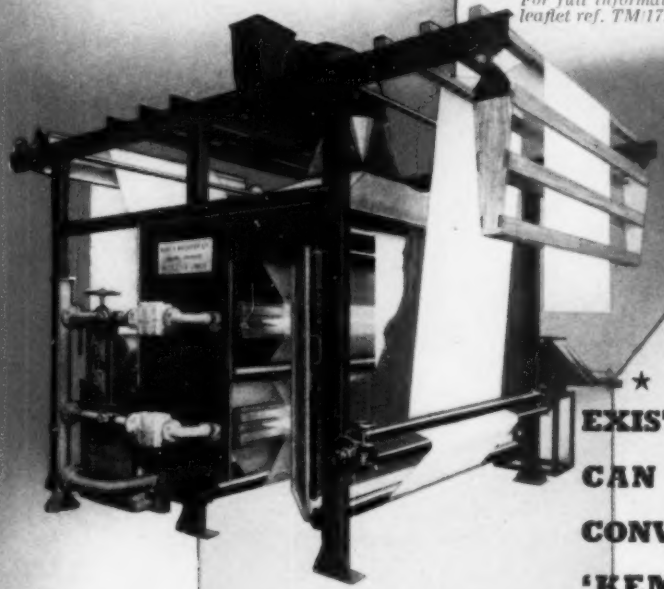

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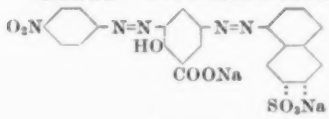
F 230 - E

Page *Colour Index No.*

3128 *add*

20151 **C.I. Mordant Brown 90** (*Brown*) *Discoverer* — Chemapol

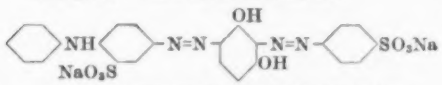


p-Nitroaniline (2) → Salicylic acid

1,6(and 1,7)-Cleve's acid (1) → Salicylic acid

3129 *add*

20175 **C.I. Acid Brown 213** *Discoverer* — Pol

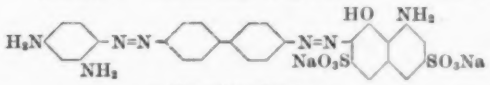


5-Amino-2-anilinobenzenesulfonic acid (1) → Resorcinol

Sulfanilic acid (2) → Resorcinol

3147 *add*

22046 **C.I. Direct Violet 88** *Discoverer* — Pol



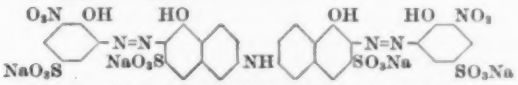
Benzidine → *m*-Phenylenediamine

Benzidine → (*Alk.*) H acid

3249 *add*

29128 **C.I. Direct Blue 184** *Discoverer* — Pol

A copper complex derived from



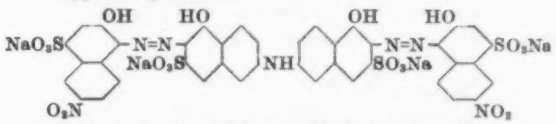
2-Amino-6-nitro-1-phenol-4-sulfonic acid (2 mol.)
⇉ 6,6′-Iminobis-1-naphthol-3-sulfonic acid;

and convert to the copper complex

add

29130 **C.I. Direct Blue 185** *Discoverer* — Pol

A copper complex derived from



1-Amino-6-nitro-2-naphthol-4-sulfonic acid (2 mol.)
⇉ 6,6′-Iminobis-1-naphthol-3-sulfonic acid;

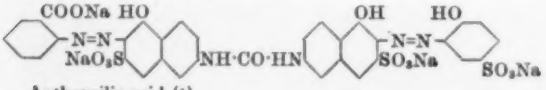
and convert to the copper complex

Page *Colour Index No.*

3249 *add*

29167 **C.I. Direct Red 99** (*Bluish red → Bordeaux*)

Copper complex derived from



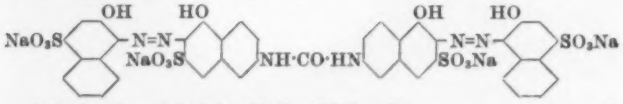
Anthranilic acid (1) →
6,6′-Ureylenebis-1-naphthol-3-sulfonic acid
2-Amino-1-phenol-4-sulfonic acid (2) →

and convert to the copper complex

3251 *add*

29232 **C.I. Direct Violet 87** *Discoverer* — Pol

A copper complex derived from

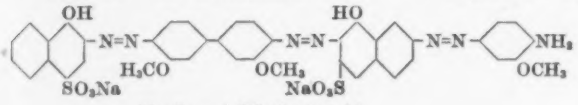


1-Amino-2-naphthol-4-sulfonic acid (2 mol.)
⇄ 6,6′-Ureylenebis-1-naphthol-3-sulfonic acid;

and convert to the copper complex

3283 *add*

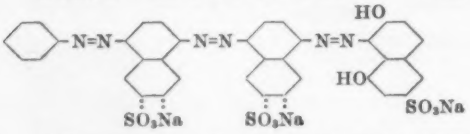
31951 **C.I. Direct Blue 183** *Discoverer* — Pol



Nevile and Winther's acid →
o-Dianisidine
(*alk.*) Gamma acid → *o*-Anisidine

3293 *add*

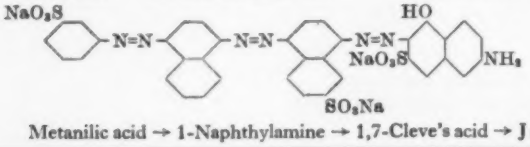
34100 **C.I. Direct Black 101** *Discoverer* — Pol



Aniline → 1,6(and 1,7)-Cleve's acid → 1,6(and 1,7)-Cleve's acid
→ 4,6-Dihydroxy-2-naphthalenesulfonic acid

add

34139 **C.I. Direct Blue 186** *Discoverer* — Pol

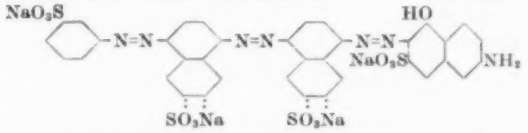


Metanilic acid → 1-Naphthylamine → 1,7-Cleve's acid → J acid

Page *Colour Index No.*

3294 *add*

34146 **C.I. Direct Blue 187** *Discoverer* — Pol

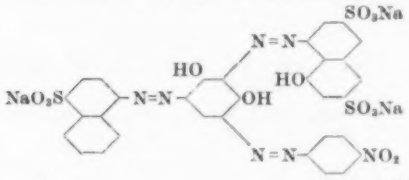


Metanilic acid → 1,6(and 1,7)-Cleve's acid → 1,6(and 1,7)-Cleve's acid → J acid

3298 *add*

34907 **C.I. Acid Brown 214** *Discoverer* — Pol

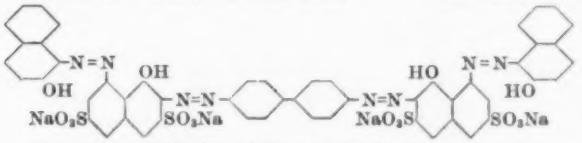
A mixture of azo dyes possibly including



[Naphthionic acid (1) → Resorcinol] ←(3) H acid
←(3) *p*-Nitroaniline

3305 *add*

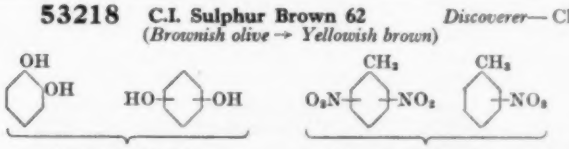
35415 **C.I. Direct Black 100** *Discoverer* — Pol



Benzidine ⇉ (*alk.*) H acid (2 mol.) ⇉ 2-Naphthol (2 mol.)

3447 *add*

53218 **C.I. Sulphur Brown 62** *Discoverer*— Chemapol
(*Brownish olive → Yellowish brown*)



Catechol and other bivalent phenols

Residue from manufacture of Dinitrotoluene

Heat these intermediates with sodium tetrasulfide

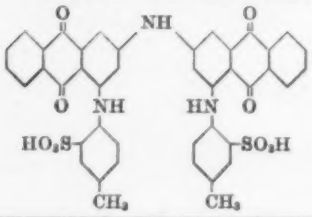
3488 *add*

59856 **C.I. Vat Blue 51**

Treat violanthrone with half the quantity of hydroxylamine used in the preparation of C.I.59855

Page	*Colour Index No.*
3513	*add*

65008 **C.I. Acid Black 97** (*Greenish grey*) *Discoverer* — Chemapol



VOLUME 4

Amendments

Page							
4004		*add*	Stu	John & E. Sturge Ltd., Birmingham, England			
4008		*add*		Sturge, John & E. Ltd., Birmingham, England (Stu)			
4162	for Brenthol ITR	ICI					
	read Brenthol LTR	ICI					
4170	for Calcocid Alizarine Violet NC	CCC					
	read Calcocid Alizarine Violet RNC	CCC					
4171	for Calcocid Ink Blue R ...	CCC	C.I. Acid Blue 93	... ...	1271	42780	
	read Calcocid Ink Blue R ...	CCC	C.I. Acid Blue 22	... ...	1239	42755	
4173	for Calcoloid Brown RRP ...	CCC					
	read Calcoloid Brown RRWP ...	CCC					
	for Calcomine Black G	CCC					
	read Calcomine Black EXL ...	CCC					
	for Calcomine Dark Blue B ...	CCC					
	read Calcomine Dark Blue BN ...	CCC					
4204	for Cibanone Olive 2B	CAC, Ciba					
	read Cibanone Olive FB	CAC, Ciba					
4248	for Direct Supra Light Violet MRL	ICS	C.I. Direct Violet 55	... ...	2159	17515	
	read Direct Supra Light Violet MRL	ICS	C.I. Direct Violet 46	... ...	2156	17515	
4269	for Fast Light Red BG	YDC					
	read Fast Light Red GL... ...	YDC					
4334	*beneath* Lake Orange GR	IG					
	add Lake Orange RO	Pol	C.I. Acid Orange 8	... ...	1057	15575 (Ba Lake)	
	for Lake Red RBKK	Pol					
	read Lake Red RBKX	Pol					
	for Lake Red RCKK	Pol					
	read Lake Red RCKX	Pol					
	beneath Lake Yellow	NAC					
	add Lake Yellow SWN	Pol	C.I. Acid Yellow 1	... ...	1003	10316 (Ba Lake)	
4463	for Tertrochrome Black A ...	CT					
	read Tertrochrome Black GA ...	CT					
4464	for Tertrodirect Black V ...	CT	C.I. Direct Black 28	... ...	2335	35260	
	read Tertrodirect Black V ...	CT	C.I. Direct Black 19	... ...	2332	35255	
	for Tertrodirect Brown CBO ...	CT					
	read Tertrodirect Brown CB ...	CT					
4465	for Tertrodirect Pink RD ...	CT					
	read Tertrodirect Pink DR ...	CT					
4466	for Tertroxane Orange R ...	CT					
	read Tertroxane Orange RN ...	CT					
4568	in 2504 under **C.I. Vat Blue 20**						
	delete add **Monolite Blue 3R ... ICI**						
4576	3053 for *add* **14058 Mordant Yellow** *read*						
	add **14058 C.I. Mordant Yellow 60**						
4577	3106 for *add* **18092 Mordant Green** *read*						
	add **18092 C.I. Mordant Green 52**						
4579	3564 for *add* **73685 Vat Green** *read*						
	add **73685 C.I. Vat Green 25** (*Green*)						

New Books and Publications

Deutscher Färberkalender 1959

(Volume 63)

Edited by O. Mecheels. Pp. iv + 64 + vi + 432 + 64 + 64. Munich: Franz Eder Verlag. Price, DM 13.50 ($3.50).

If the current year book differs little from its recent predecessors in its form and general layout, that does not necessarily indicate a resistance to change. This work has been reproduced annually for some considerable time, certainly for a sufficiently long period to enable matter of little value to be bred out. Particularly in the case of a work of reference—partial or entire—it is advantageous to be able to tread familiar ground without constant reference to an index.

The 1959 volume is still reasonably small. It is surprising how much information the editor has contrived to include and still allow room for a diary and a large number of advertisements. The major part of the space is occupied by some 28 monographs. These reflect the current interest in man-made fibres, with which topic about one-half are concerned. The ever present difficulty of static charge on these fibres and the prevention of its development are discussed, as are the methods available for the waterproofing of polyamide fibres. There is a paper on present-day American practice in the dyeing of man-made fibres, and a more theoretical discussion of the mechanism of the absorption of copper ions in the dyeing of polyacrylonitrile fibres. The dyeing of bulked yarns of Orlon and Dralon is also described.

Mixtures of man-made and natural fibres are another important topic covered by the monographs, papers being devoted to white effects in cellulose triacetate fancies and the dyeing of polyacrylonitrile–cotton blends and mixtures of polyester (Trevira) fibres with wool and with cotton.

The remaining monographs cover a miscellany of subjects: finishing is represented by papers on the chemical finishing of wool and of silk; there are articles on the continuous dyeing of cotton with direct dyes, the application of Cibacron dyes to wool, and one-bath chrome-dyeing operations on the same fibre. Ultraviolet absorbers are discussed in an interesting paper, and a number of patents granted during recent months are reviewed. The range of interests covered, therefore, is wide, and this section of the book is worthy of the attention of those who are concerned with a wide field in dyeing and finishing.

The remainder of the work is made up of reference sections. These include a very comprehensive table of fully synthetic fibres and an impressive list of fibres manufactured in Western Germany. A long section is devoted to new dyes and speciality chemicals (in which figures, incidentally, a novel polyphosphate known as "Calgon"). A fairly extensive buyer's guide is included, as is the usual section on chemicals employed in dyeing and finishing.

The publishers of this work are affording a very valuable service to the German dyeing trade. One could wish for an English equivalent.

S. R. Cockett

Paint Film Defects

Their Causes and Cure

By Manfred Hess. London: Chapman & Hall Ltd. 1958. Pp. xvi + 544 with 87 figures. Price, 75s. 0d. ($12.00).

This book was first published in German in 1938, and the first English translation appeared in 1951. The latter was reprinted in 1952, and the present volume is described as "reprinted with revisions". The book is designed as a work of reference, and is practical rather than scientific in its approach, so that it should be useful to the paint user as well as to the technologist in the manufacturing or raw-materials laboratory. The book does not lend itself to continuous reading, being, in effect, a dictionary of paint faults, so that widely differing types of material are brought together under the heading of a defect which, although having the same name, may be different in nature in the different paints.

Nomenclature is, indeed, a basic difficulty in compiling a work such as this, and individual technologists will inevitably disagree with some definitions both expressed and implied. The author is aware of this difficulty, and prints as a "Postscript" a glossary of terms culled from various sources, including the American Society for Testing Materials, the British Standards Institution, and the Deutscher Normenausschuss (D.I.N.). This gallant attempt throws some light on dark places but points more sharply the need for the paint industry itself to draw up an agreed and definitive glossary.

The book is admirably indexed and cross-referenced to enable the user to trace a fault through the maze of possible sources. Those not versed in paint technology may be appalled by the list of faults possible—the subject index runs to 84 pages of double-column close print—and wonder that any paint film is satisfactory in practice. That the majority are so evidently speaks well for the skill of both paint manufacturer and paint user. Apparently colour may be a major source of trouble. There are 44 entries in the index under "pigment", but of six entries under "fading" four are due to factors independent of the pigment itself.

Paint manufacture, in common with other technologies, has advanced enormously during the twenty years since this book was first published, but even now it is by no means an exact science. Paint formulation, like paint application, remains in many respects an acquired skill, depending on practice and experience as well as on basic scientific knowledge. This being so, opinions vary on the causes of many of the ills to which paint is liable and equally on the steps necessary to avoid the trouble or to effect a cure, so that many statements

can be found with which a particular reader will disagree. For example: "Most correctly compounded air drying white synthetic resin enamels or nitrocellulose lacquer films practically never yellow even in the dark" (p. 352)— if this is true, few "correctly compounded" white enamels appear on the market!

But such discrepancies are inevitable in the present state of knowledge, especially in a book covering so wide a range of published data in addition to its author's own experience. Used with care, *Paint Film Defects* should undoubtedly be of enhanced value in this new reprint and at least suggest lines of attack on any particular problem if not giving a complete answer to it.

R. B. WATERS

The Chemistry and Technology of Leather Volume 2—Types of Tannages

Edited by Fred O'Flaherty, William T. Roddy, and Robert M. Lollar. *American Chemical Society Monograph Series* No. 134. Pp. xiv + 554. New York: Reinhold Publishing Corpn. (London: Chapman & Hall Ltd.) 1958. Price, 132s. 0d.

This is the second of a series of four volumes published in the A.C.S. Monograph series. It is the work of sixteen authorities, eleven American, the others being from Great Britain, France, Western Germany, South Africa, and India. Volume 1 covered "Preparation for Tannage", and the present work deals largely with the chemistry and properties of the main tanning materials.

In the two introductory chapters, the physical and chemical changes occurring in the production of leather are examined, and the measurement of shrinkage phenomena is related to the theory and practice of tanning.

Aldehyde tannage, with particular reference to formaldehyde, is covered in a thirty-page chapter, half of which is a survey of the evidence for the combination of formaldehyde with reactive centres, followed by brief practical tanning details.

The chemistry of the vegetable tannins is ably covered, and this chapter clearly sets out how chromatography and other techniques have helped to resolve the complex mixtures which constitute vegetable extracts. After a section on their physical chemistry, with special reference to particle size, there is a short but informative review of the non-leather applications of tannins. A forty-page chapter on the process of vegetable tannage gives much simple, and already widely published, detail in a survey of the individual tannins before giving factors influencing vegetable tannage and general methods of vegetable tanning. The writer on the mechanism of vegetable tannage suggests that, although the chemical process of tannage is characterised by multipoint hydrogen bonding of the phenolic tannin to the peptide linkage of the collagen, the non-specific deposition of tannin in the interstices of the collagen fabric contributes materially to the commercial properties of the leather.

The following three chapters cover the chemistry, practice, and mechanism of chrome tannage, and together with the corresponding sections on vegetable tannage occupy nearly half of the volume. When dealing with the chemistry of chrome tanning, the author could have assumed more knowledge of chemistry in a book which the introduction states to be written for chemists. Like the corresponding chapter on vegetable tanning, that on the practice of chrome tanning gives the impression of being padded out with established information and contributes little to a volume of this standing. The photographs of the inside of a tannery are quite useless. In a very full chapter on the mechanism of chrome tanning, the various theories, and also the factors influencing chrome tannage, are competently reviewed, though somewhat coloured by the author's own views.

The existing information on zirconium tanning is clearly presented, as are the limited data available on aluminium, iron, polyphosphate, and silica tanning.

Synthetic tannins enjoy a section to themselves, although this deals only with the replacement tannins, the auxiliary syntans having a very brief and incongruous mention in an earlier section on vegetable tannins. After a review of the earlier literature on the basic chemistry of the synthetic tannins, most of the chapter is devoted to German and American materials. This is understandable, since the constitutions of the former are available in the German Industry Reports (BIOS 762), and the latter are those of which the author has most experience. The difficult, and at present unresolved, problem of the evaluation of syntans is fairly fully discussed.

Related to syntans and of increasing commercial importance are the resin tannins. More information on the use of these materials on chrome retanning would have been welcomed.

The somewhat empirical oil tannage and the comparable sulphonyl chloride tannage are coupled together, and there is full coverage of the testing and the properties of oils suitable for tanning.

The chapter on combination tannages deals largely with army retan upper leather. A lengthy history is followed by some outline practical methods, and various retanning agents, e.g. resins, are mentioned. The use of product names, measures, and expressions more common to the American reader greatly reduces the value of this chapter to those not fully familiar with leather terminology. A more complete and fundamental appraisal of the retanning of chrome leather would have been most valuable. Finally, the stability of leather is considered; and atmospheric, perspiration, and bacterial, etc. attack are very fully covered, and much useful information is collated.

The title of this volume would suggest that it contains more practical information, but its main value will be to the chemist and the technologist. Such practical details as are given are generally

quite brief and are conspicuously different in quality of presentation from the more theoretical sections. Such criticism should perhaps be withheld until the next volume appears, the chapter headings of which suggest that it will be mainly practical, but the present volume gives the impression that it would benefit from some rearrangement and more vigorous editing of some sections.

This is, however, a reference book and as such will be very valuable. It contains much information that would otherwise be difficult to find, and there will be few questions on most aspects of modern tanning materials which it does not help to answer. A very full bibliography of over 1,500 references and an adequate index are valuable aids for the serious scientific reader, for whom the book is primarily written.

J. HUSSELBY

Wollschädlinge und ihre moderne Bekämpfung

By A. Herfs and H. Stötter. Pp. 78 with numerous illustrations. Leverkusen: Farbenfabriken Bayer AG. [1958.] No price.

In the first 54 pages of this book Dr. Herfs, the former head of the Textile Zoological Laboratory at Leverkusen, gives a concise overall account of the insects that are liable to attack wool. It contains little that is new, but nevertheless is a most interesting and well illustrated account well suited to the needs of anyone coming new to the subject. It is, however, the history of the development of the Eulans from 1922 to the present day, written by Dr. Stötter, for so many years head of the Eulan Technical Department at Leverkusen, that will receive most attention from members of the Society, disclosing as it does the constitutions of the early inorganic Eulans and of the organic Eulans RHF, N, CNA, FL, NK, BL, BLN, and WA extra conc. The history of the Eulans, especially from Meckbach's original idea to the successful launching of Eulan N in 1934, is a romance of industry, but, as in most accounts of this kind, Dr. Stötter makes the work of discovery and development look vastly more easy and simple than it really was. This is because only the successful products are mentioned, and the manifold difficulties and disappointments and the tedious labour involved that beset the development of mothproofing both in the Bayer Co.'s laboratories and in their customers' works during the first dozen years of its existence are not even hinted at. There are sound commercial reasons for this, for it is unreasonable to expect a firm to publish information which conceivably might still be of use to competitors. Nevertheless, there is no doubt that an account of the work that led into blind alleys or revealed unsuspected difficulties would provide a valuable lesson to young chemists commencing work on new developments quite outside the field of mothproofing. We are much indebted to Dr. Stötter for this lucid account of the successful results of his colleagues' and his own work, but is it too much to hope that he will write an account of the difficulties and disappointments they met with— the Eulan B of the later 1920s is a case in point— so that it can be kept in the archives of the Bayer Co. until such time as commercial considerations permit it to be made available for the instruction of a new generation?

C. O. CLARK

Fulling Mills

A Study in the Application of Water Power to the Woollen Industry

By R. A. Pelham. Booklet No. 5 of the Wind and Watermill Section of the Society for the Protection of Ancient Buildings. Pp. 16 + 4 plates. London: Society for the Protection of Ancient Buildings. 1958. Price (stiff paper cover), 3s. 0d.

Fulling mills have been in use in Britain for over 750 years, the earliest recorded examples being in 1185 at Newnsham, Yorkshire, and Burton in the Cotswolds, though since the invention of the cylinder milling machine by John Dyer of Trowbridge in 1833 their use has gradually declined, so that it is in few places that they can be found in active use today. This book summarises their history, particularly as regards the effect their introduction had on the organisation of industry and on the siting of many modern mills and towns. Thus the author points out that I.C.I.'s works at Witton, Birmingham, have an industrial pedigree going back in a direct line to a fulling mill that was set up on the banks of the River Tame in the fourteenth century. For all who desire to learn about the history of what was and still is one of our most important trades, both for home consumption and for export, this booklet is strongly recommended.

C. O. CLARK

Principles of Cotton Printing

By D. G. Kale. Pp. xix + 452 + xxii plates. Ahmedabad, Bombay State: The Publication Committee for Professor D. G. Kale's Book on Cotton Printing. [1957.] Price, rupees 25.

The author divides his subject into four sections— I (pp. 1–100) *Methods of Printing*, II (pp. 101–130) *Preparing the Cloth for Printing* and *Thickening Agents*, III (pp. 131–414) *Styles of Printing*, and IV (pp. 414–431) *Aftertreatment and Finishing of Printed Goods*. Addenda (pp. 435–440) mainly concern Procion dyes and eleven corrections.

Section I deals with the older hand-printing methods. An interesting account of stencil printing, which was introduced by the Japanese, is followed by a full account of screen printing and its development, including screen preparation and hand and mechanical screen printing up to the introduction of the Buser machine and the Zimmer duplex machine. Roller printing and engraving are well described, and in the case of several diagrams taken from Knecht and Fothergill's *Textile Printing* only the latter is acknowledged, to the omission of Dr. Knecht, a former editor of this *Journal*. In referring to Bell's invention of roller printing, the author gives the date as 1733

in his Introduction (p. xii) and as 1785 on p. 66, whereas Bell's first patent (No. 1378) is dated 1783 and his second patent (No. 1443) 1784, neither patent being quite so complete as is shown by Fig. 48 on p. 66. Classic references are often omitted; under the description of embossed blankets (p. 74) no reference is made to the Dewey and Almey work or patents. Later in the book "Vatsols" announces the Indigosols, and whilst "Kalb's compound", presumably Kalle's indigo salt, receives mention, Bader and Sunder are not named as inventors of Indigosol O. It is interesting to note (p. 78) that, presumably, in India the keyway in the mandrel and the key or tab are dispensed with in roller printing, and only "the tapering shape itself serves to secure a tight fit non-slip grip between the mandrel and the hollow design roller". Out of a number of spelling mistakes throughout the book perhaps the most obvious is "Bruin" (p. 82) for the name of the hand engraver's tool, the *burin*, and the Scots would frown on "Ayer" (p. 82) for *Ayr*; later "John Thomas of Mayfield" (p. 423) should read *John Thom of Mayfield, Manchester.*

Section II curiously combines preparation of the cloth for printing (largely bleaching) and thickening agents for colour making. The chlorine and recent hydrogen peroxide bleaches (du Pont, Becco, etc.) are dealt with. The lime bleach given for the "dyed style" includes three boils—(1) lime, (2) resin, (3) soda ash (p. 114). The two latter have been combined for a very long time.

Section III, which is the largest, deals with discharge and resist printing, metal printing, flock printing, and crimping in addition to topical printing. In this larger section mis-statements and curious explanations abound, of which the following examples are but a few. In a description of the open soaper (p. 148) the sentence "It is just like a jigger working" is erroneous. In a great number of recipes "gum" or "thickening" is given without further description. Some evidence for the statement (p. 156) that the alumina–arsenic–basic dye lakes are non-toxic would be interesting. On concluding a short paragraph on "Flash Ageing" it is stated (p. 199): "No special steaming arrangement is necessary. Ordinary box with a steam pipe will also serve the purpose", whereas in fact much thought and work have been given to the design of this plant. The simile used to explain the difference between the discharge and resist style (p. 329) is "The maxim 'prevention is better than cure' . . . . . Quinine prevents attack of malaria while diphortic mixture brings down the fever temperature which is already on."

The printed patterns on pp. 144, 155, 311, and 395 are poor productions, and should not have been mounted.

The book is interesting in presenting an Eastern viewpoint on the subject of printing, and records interesting accounts of Eastern, particularly Indian methods, viz. the Dhobi Bhatti bleaching, Eastern madder style, the Ram Nam process, Indian hank printing machine, khadi or tinsel printing, washing prints in the Ganges, etc. Published by subscription, it is very cheap, and the publication committee are remiss in not rendering more assistance to the author in proofreading, etc.

J. G. HURST

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

American Cyanamid Co.

DYES TECHNICAL BULLETIN No. 852. *Printing with Emulsions*— This is a reprint of a paper by R. D. Greene, *Amer. Dyestuff Rep.*, **47**, P 191 (1958).

Ciba Ltd.

CIBALAN BLACKS 2GL: 2BL— These neutral-dyeing metal-complex dyes are very similar to Cibalan Black BGL in dyeing behaviour and fastness characteristics, differing only in hue, the 2GL brand giving greenish blacks and the 2BL brand bluish blacks, and the hue in both cases being substantially unaltered in artificial light. They are of interest for dyeing wool, polyamide fibres, and silk, and mixtures of the two latter with wool and for mélange printing. The card includes dyeings of the BGL brand and fastness data for comparison. Fastness figures on wool include— *Black 2GL*: Light 7–8, alkaline milling ECE b 4, washing ECE b 4. *Black 2BL*: Light 7–8, alkaline milling ECE b 4–5, washing ECE b 4–5.

CIBALAN BRILLIANT SCARLET RL— This neutral-dyeing dye gives bright scarlets on wool, silk, and polyamide fibres. Like the first three members of the Cibalan Brilliant range, and unlike the other Cibalans, it is not a metal complex but contains reactive groups in the dye molecule through which covalent dye–fibre linkages are formed. Since the dyeing behaviour of these two groups of dyes and the fastness properties of the dyeings are very similar, the Cibalan Brilliant range are suitable not only for use alone and in mixtures with each other but also for brightening Cibalan dyeings. Brilliant Scarlet RL gives solid dyeings on wool–polyamide fibre mixtures and is also suitable for mélange and direct printing. Fastness figures on wool include— Light 6, washing ECE b 4–5, alkaline milling ECE b 4G.

CIBALAN RED BROWN RL— This neutral-dyeing metal-complex dye gives bright reddish browns on wool, silk, and polyamide fibres. It is recommended for use alone and as the red component in mixtures with other members of this range, particularly where maximum fastness to light is required. Fastness figures on wool include— Light 6–7, washing ECE b 4–5, alkaline milling ECE b 5.

CIBANONE YELLOW FLGR— This vat dye gives bright reddish yellows on cellulosic fibres. It is recommended for use alone and in mixtures on yarn for coloured woven cloths and furnishings. Resin finishes based on Urcol P or Lyofix CH do not impair light fastness and produce only a very slight change in hue. Solid dyeings are obtained on cotton–viscose rayon mixtures. Fastness figures on cotton include— Light 6–7, soda boiling ECE b 4, chlorine ECE b 4–5, peroxide bleach ECE 1 5.

CATALYSER PR CIBA— A catalyst for thermosetting resins, in particular Lyofixes PR and CH.

STABILISER VP CIBA— An assistant for use when applying vat dyes by prepigmentation methods. It promotes penetration of yarn packages and exhaustion of the

pigmenting liquor and inhibits migration during dyeing when pad–dyeing cloth.

UVITEX A— A fluorescent brightening agent for acrylic staple fibre. It gives a neutral white of good fastness to light and washing and can be applied in acid sodium chlorite bleach baths.

Farbenfabriken Bayer AG.

ACRAMIN ORANGE F5G— This pigment for resin bonding gives bright yellowish oranges and is primarily intended for full orange prints. It may be applied alone and in combination with the other dyes of this range, using Acramin F, Acramin FD, and Acramin SL binders. Fastness figures for a machine print fixed by dry heat and then stored for 12 hr. to complete the fixation include— Light 6, washing (100°C.) 4–5, chlorine (a) 4–5, trichloroethylene (test carried out without rubbing) 2–3.

INDANTHREN BRILLIANT SCARLET EFR— This vat dye gives bright scarlets on cellulosic fibres. Dyeings and prints have very good fastness to light and wet treatments including soda-boiling and chlorine and peroxide bleaching. Curtains, furnishings, and all materials required to withstand repeated laundering dyed with it may carry the Indanthren label irrespective of depth of colour. Fastness figures on cotton include— Light 6–7, washing (c) (203°F.) 4–5, soda-boiling (b) 4, chlorine (b) 4–5.

BAYER-COLORIST SPECIAL EDITION— This special edition (No. 7) is devoted entirely to describing the properties and applications in pad–dyeing and printing of Naphtol AS–FGGR, an azoic coupling component based on a phthalocyanine derivative and designed to give a wide variety of bright greens in combination with a number of azoic diazo components.

Farbwerke Hoechst AG.

IMPERON K DYESTUFFS FOR PIGMENT PRINTING— This card contains prints in three depths on cotton cloth of 8 Imperon K dyes for pigment printing. They are free from synthetic resin and thus may be applied in conjunction with practically all binding agents of the oil-in-water type. The actual pigments are the same as those present in the original Imperon range, the colour strength of the new brands ranging from 2½ to 3 times that of the older type. The hue and fastness characteristics of prints of the K brands are very similar to those produced with the earlier range as far as this depends on the pigments themselves.

J. R. Geigy AG.

IRGALAN BLUE FGL— This neutral-dyeing metal-complex dye gives blues which are greener and brighter in hue than those obtainable with the Blue GL brand. In dyeing behaviour and fastness characteristics it is completely compatible with other members of the range. In addition to its use on loose wool, slubbing, yarn, and cloth, it is also of interest for dyeing silk and polyamide fibres and in printing. Fastness figures on wool include— Light 6–7, alkaline milling (b) 4, acid chlorination (0·5 g. active chlorine per litre) 4–5.

IRGALAN BORDEAUX GL— This neutral-dyeing metal-complex dye gives bordeaux, yellower in hue than those obtainable with the 2BL brand, and is particularly suitable for use in combination with Irgalan Rubine RL. In addition to its use on wool, it is of interest for dyeing silk and polyamide fibres and for printing. Fastness figures on wool include— Light 7, severe milling 5, washing (b) 5.

IRGANOL YELLOW 4GLS— This neutral-dyeing acid dye gives bright yellows on wool practically identical in hue with those obtained with Irganol Yellow 5GLS but superior in fastness to light. Its low rate of exhaustion makes it more level dyeing and ensures better penetration. In conjunction with Irganol Orange GRLS and Irganol Brilliant Green GLS a range of bright dyeings of excellent fastness can be produced which are of particular interest for swimwear and sportswear. Fastness figures on wool include— Light 6–7, washing (b) 4–5, sea water 4–5.

The Geigy Co. Ltd.

FAST TO LIGHT ACID DYES ON WOOL PIECE GOODS— This card contains dyeings in five depths on wool gabardine cloth of 15 acid dyes selected for their ability to meet the high standard of fastness to light and perspiration demanded on such material. For maximum fastness and ease of application Erio Fast Brown 5GL, Erio Fast Red 2BLS, and Erio Anthracene Brilliant Blue 4GL are outstanding. Twelve dyeings representing typical greys, fawns, and stones, produced with this combination, are included together with 12 others based on Erio Fast Yellow 3GS, Erio Fast Rubine 3GS, and Erio Anthracene Brilliant Blue 2GL, a combination recommended when fastness requirements are less exacting or when particularly bright colours are required.

TINOLITE COLOURS— This card contains prints in two depths on cotton of 20 Tinolite pigment emulsions for application by resin-bonding to the fibre to all types of material, including man-made and inorganic fibres. For application the pigment paste is combined with an oil-in-water resin binder (Tinolite Binder CM), printed on to the material using either screen or roller, dried, and dry-heat-cured, preferably for 1–4 min. at 300°F. No further treatment is necessary, but in high-class printing on certain types of material a light soaping may be desirable to remove any soluble components of the printing paste. Matt whites may be obtained with Tinolite White Printing Paste. For printing with metallic powders Tinolite Printing Binder GW is recommended. The colour range is complete, and each member can be mixed in all proportions with any other. The card is loose-leaf, each colour being given a page to itself, and the patterns are mounted on a dark grey backing for ease of examination and comparison of colours.

Imperial Chemical Industries Ltd.

QF CALEDON PRINTING YELLOW GN— This vat dye gives bright golden yellows of excellent fastness to light when printed on cellulosic fibres. Oxidation should follow steaming as rapidly as possible to avoid production of a greenish product which can be oxidised further only with difficulty. If this trouble occurs, a weak dry chemick may brighten the colour. Fastness figures on cotton include— Light 6, washing (repeated severe, 5 times at 100°C.) 4–5.

CARBOLAN RUBINE 2B— This neutral-dyeing acid dye gives bright bluish reds on wool and polyamide fibres, being particularly useful on the former for deep reds. The wet-fastness of dyeings on polyamide fibres can be still further improved by "back-tanning". It is suitable for direct printing on wool, silk, and viscose rayon (by the urea process). Dyed grounds on wool, silk, and nylon may be discharged to a moderate white using Redusol Z. Fastness figures on wool include— Light (Wilmslow) 4–5, (Bombay) 5, alkaline milling 4, chlorination (acid) 5.

SQ CALEDON GRAINS— The following vat dyes of the Caledon range are now available as SQ ("superfine quality") Grains— Gold Orange 3G, Dark Brown 6R, Brilliant Red 6B, Blue XRC, and Khaki 2G. They are marketed in granular form, show little tendency to dust or "fly", and are easy to handle in weighing. They disperse quickly in water to give dispersions of very small particle size free from large particles which would lead to specking or form nuclei for the aggregation of smaller particles. They show little tendency to migrate during the drying of pigment-padded goods, and reduce readily and give high colour yields in continuous processes based on "flash" reduction, e.g. pad–steam and pad–Standfast methods. They can be used in any printing application for which the existing FD Powder Fine brands are used at present.

CAROMASTRAL GREEN H-MS— A coprecipitated blend of a lead chrome pigment and a modified phthalocyanine blue, similar in hue and strength but controlled to contain less than 5% of soluble lead (according to *B.S.* 282 : 1953, Appendix K). It is recommended for use in all types of paints but is of particular interest for industrial finishes when spray application is used.

QF DURINDONE PRINTING ORANGE R— This vat dye gives bright oranges on cellulosic materials. Ease of fixation makes it of value in both print-on and discharge styles. When used as an illuminating colour in discharge printing, no Caledon Developer AQ Paste should be added. Fastness

figures on cotton include— Light 4, washing (repeated severe, 5 times at 100°C.) 3–4, hypochlorite ("dry-chemick") 5.

MONASTRAL FAST BLUE BXS— This is a pure pigment of outstanding brilliance of hue, high tinctorial strength, and excellent fastness properties which has been developed primarily for use in letterpress, lithographic, and other inks based on drying oils. Where active solvents, e.g. aromatic hydrocarbons, are present, a solvent-stable LBX brand has been developed.

MODE SHADES ON ACRILAN LOOSE FIBRE, SLUBBING AND YARN— This card contains 18 dyeings on Acrilan slubbing produced with either single dyes or mixtures from the following classes— level-dyeing acid, fast-to-milling acid, disperse, chrome, acid-dyeing metal-complex, reactive (Procion Brilliant Blue H7G), and azoic. The text includes notes on selection of dyes, details of suitable dyeing methods, and fastness data.

ALCIAN DYES DYED ON COTTON, LINEN AND VISCOSE RAYON— This card contains dyeings on cotton, viscose rayon, and linen cloth and on cotton yarn of the seven members of the Alcian X range. These dyes may be applied to cloth on the winch or jig or by padding and to yarn in circulating-liquor machines, the general procedure being to apply the dye to the fibre from a dyebath set at pH 3·5–4·0 with acetic acid and then to insolubilise the dye by treating the material at the boil in a solution of sodium carbonate. Alcian Blue 8GX gives brilliant turquoise blues, and the other blues also can be applied satisfactorily, either alone or in mixtures. The Greens 2GX and 3BX are less satisfactory, as reproducibility of hue may be difficult to obtain. Similarly, mixtures of Yellow GX and the blues are not recommended, although the Yellow GX alone may be used for golden yellows. Irregularities in the dyeing properties of the fibres or cloth construction are not always completely covered in pale depths, particularly with viscose rayon. Complete stripping of dyeings on cellulosic fibres is possible, using a caustic soda–hydrosulphite strip in presence of Lissolamine A 50%, with Blues 7GX and 8GX. Blues 2GX and 5GX can be reduced in depth, the greens can be reduced to a yellow, but Yellow GX can be only slightly reduced in depth.

PROCION DYESTUFFS FOR SPUN VISCOSE RAYON SHIRTINGS— This card contains 14 dyeings on 100% spun viscose rayon cloth produced with Procion Yellow R, Rubine B, and Brilliant Blue R or mixtures of them using the pad (dye + bicarbonate) → dry → wash method. None has a light fastness (daylight) less than 5 or a fastness of less than 5 to one wash at 85°C. after a resin crease-resist finish followed by a soaping treatment.

MODE SHADES ON TERYLENE–WOOL UNIONS— This card contains 20 dyeings on cloth woven from a 50:50 blended yarn of Terylene staple fibre and wool. The Terylene component has been dyed with mixtures of disperse dyes in all cases. Dyes used for the wool include fast-to-milling acid, chrome, and acid-dyeing metal-complex dyes and, in two instances in both of which a single-bath method was used, a mixture of Propolan Red 3G and Solway Blue 2G. In the two-bath dyeing procedure the disperse dyes were applied first in presence of Tumescal OP, followed by a clearing treatment with ammonia and hydrosulphite, the wool being dyed in a fresh bath. Reduction of the disperse dye stain on the wool to a minimum is essential for maximum fastness. This may be achieved by using the procedure recommended and maintaining as high a dyeing temperature as possible, e.g. by using an enclosed winch when dyeing cloth to deep colours.

TECHNICAL INFORMATION LEAFLETS— *Dyehouse No. 446. Dyestuffs for Leather. Assessment of Penetration; Solubility; Fastness to Light, Washing and Water*— This leaflet includes all the previously published data together with details of a revised washing test, figures for the fastness to water of dyeings on mordant chrome gloving leather, and fastness assessments of the Procion reactive dyes which are now available.

Dyehouse No. 450 (replaces No. 400). *Subject Index to Technical Information Leaflets (Dyehouse) No. 161–449.*

Dyehouse No. 453. Viscose Rayon: Processing Aids— These are listed, and details are given for their use when washing, bleaching, lubricating, softening, and scrooping filament and staple yarns.

Dyehouse No. 460. Procion Dyestuffs— Weathering Fastness— It has been shown that dyeings or prints of the Procion and Procion H dyes on cotton and linen cannot be classified as "suitable" when subjected to outdoor exposure; i.e. they cannot be rated 6 or higher against the light fastness standards exposed simultaneously but behind glass. Procion Brilliant Yellow 6G, Brilliant Yellow H5G, Yellow R, and Yellow HA are classified as "borderline". Of these, those giving greener hues are of interest, however, since they give dyeings and prints superior in many cases to those obtained with vat dyes and not subject to photochemical tendering.

Dyehouse No. 461 (replaces No. 387). *Procion Dyestuffs on Leather*— It is confirmed that the H brands are, in general, insufficiently reactive to give level dyeings of adequate fastness by any dyeing method yet devised. Brilliant Blue H7G and Black HG will give level dyeings, but these have relatively poor wet fastness. The more highly reactive members are very suitable for producing fashion colours on gloving and clothing leathers where the fastness demands are comparable with those met with on textile materials. It is noted that certain combinations are incompatible in respect of penetration and, in addition, Black HG is only recommended for use alone.

Dyehouse No. 462. 1959 Fashion Colours— Nylon Hose (The Midland Hosiery Dyers' and Finishers' Federation).

Dyehouse No. 464. Emulsion Thickenings in Textile Printing— Emulsion-thickened print pastes consist essentially of either oil-in-water or water-in-oil emulsions of inert hydrocarbon oils, the former being preferred, since they are easier to prepare, more stable, and more readily removable. The general advantages of emulsion printing systems and the printing properties of oil-in-water emulsion thickenings are outlined, and detailed instructions are given for preparing such thickenings for use with Procions, vat dyes, Alcian X dyes, and Brentogens on cellulosic fibres. Emulsion thickenings are also of interest for use with oxidation blacks and for printing nylon, secondary cellulose acetate, cellulose triacetate, and polyester fibres. If small amounts of Bedafin 16X are incorporated, emulsion-thickened discharge pastes of the type recommended for vat dyes may be used.

Dyehouse No. 465. Cleaning, Reproofing and Stiffening of Raincoats— Procedures are described based on the use of Lissapol N plus soap and soda ash for cleaning, and Dipsanil V and Calatac VB for reproofing and stiffening.

Dyehouse No. 468 (replaces No. 268). *The Dyeing of "Terylene" Polyester Fibre— Supplement No. 5. Azoic Process for Navy Blues on Loose Staple, Slubbing and Yarn*— Brenthol FO and Brentamine Fast Blue B Base are used, applied at a maximum temperature of 130°C. The light fastness of the dyeing is good provided that not less than 2% of each component is used, a full navy having a light fastness of 6. Compared with an azoic navy based on Brentamine Fast Blue B Base and Brentosyn BB, this combination requires less time and less materials, gives much better light fastness, and makes it much easier to achieve good fastness to rubbing.

Dyehouse No. 469. Pigments for Epoxy Resin–Urea–Formaldehyde Stoving Finishes.

Dyehouse No. 470. Pigments for Amine-cured Epoxy Resin Finishes.

Dyehouse No. 471. Procion Dyestuffs: Continuous Dyeing of Cellulosic Loose Fibre and Yarns— Supplement No. 1— It has been shown that it is possible both to size and to dye yarn in beam form in one operation to give the following sequence: Impregnate (dye + size) → dry (heat) → weave → desize and scour. The size used must not react with the dye; sodium alginate and Polyox WSR 35 are examples of suitable products.

REPRINTS— The following have been issued—

Effect of Penetration on Reflectance of Dyed Textile Fibres, D. A. Garrett and R. H. Peters, *J. Textile Inst.*, **47**, T 166 (1956).

The Dyeing of New Yarns and Fabrics, I. M. S. Walls, *J. Textile Inst.*, **47**, P 724 (1956).

Instrumental Control in Colour Matching, E. Atherton, *Proc. Tech. Section British Paper & Board Makers' Assocn. (Inc.)*.

The Preparation, Properties and Rheological Investigation of Thixotropic Paint Systems, D. J. Doherty and R. Hurd, *J. Oil & Col. Chem. Assocn.*, **41**, 42 (1958).

Studies of Dyeing of Nylon with Acid Dyes. II—*Kinetics of the Process*, E. Atherton and R. H. Peters, *Text. Research J.*, **26**, 497 (1956).

Fast Dyes on Cellulosic Fibres, T. Vickerstaff, *Amer. Dyestuff Rep.*, **47**, 33 (1958).

Optical Properties of Pigments in the Near Infrared, D. Taylor, *J. Oil & Col. Chem. Assocn.*, **41**, 707 (1958).

The Selection of Coloured Pigments for Industrial Finishes, I. S. Moll, presented at the Spring Conference of the Institute of Metal Finishing (April 1958).

Sandoz Products Ltd.

FAST COLOURS FOR WOOL HOSIERY— This card contains dyeings in two depths on wool yarn of 51 dyes intended to meet the increasing demand for a high standard of fastness to light and washing on hosiery yarns and fabric, the majority satisfactorily withstanding S.D.C. Washing Test No. 2. The dyes are subdivided into the following groups— (1) Xylene Fast P dyes; (2) acid dyes giving dyeings fast to S.D.C. Washing Test No. 2 and sodium perborate; (3) Lanasyn dyes; (4) a short range of acid dyes regularly used in the hosiery trade where brightness and economy are primary considerations and the highest fastness to light and washing is not essential. A range of 22 dyeings of fashion colours is also included in a separate card inserted inside the back cover.

ARTISIL YELLOW 2GL; FORON YELLOW 2GL— This disperse dye gives bright golden yellows on secondary cellulose acetate, cellulose triacetate, polyamide, and polyester fibres. It builds up well and is suitable for application in presence of carriers and above 100°C. Dyeings and prints on polyester fibres have excellent fastness to light, sublimation, heat-setting, and wet treatments. It gives very fine dispersions stable to boiling (average particle size 0·5–1 μ.) and may be applied in circulating-liquor machines. Some fastness figures on secondary cellulose acetate include— Light (daylight SNV) 6, washing (40°C. SNV) 5.

SOLAR GREEN 3LG; CUPROFIX GREEN 3LG— This direct dye gives yellowish greens on all types of cellulosic materials. Direct dyeings and prints have very good fastness to light and aftertreatment with Sandofix WE, and especially Cuprofix SL gives very good fastness to water, washing, and perspiration. Light fastness is hardly affected by crease-resist finishing. Solid dyeings are obtainable on cotton–viscose rayon mixtures and dyed grounds are dischargeable to white. Fastness figures for a direct dyeing on viscose rayon include— Light (daylight ISO) 7, perspiration (ECE) 4, washing (40°C. ISO) 4–5.

SOLAR SCARLET RL— This direct dye gives bright scarlets on all types of cellulosic fibres. Dyeings have good fastness to light and good fastness to washing when aftertreated with Sandofix WE. The light fastness of dyeings is only slightly affected by crease-resist finishing, particularly when the Finish EN process is used. Type ACS reserves secondary cellulose acetate, and good white discharges are obtainable on cotton under both neutral and alkaline conditions. Fastness figures on cotton include— Light (daylight SNV) 5–6, washing (40°C. SNV) 2–3, perspiration (SNV) 2.

SANDOTHRENE BLACK F-N3BA— This vat dye gives dark greens of medium fastness to light when dyed direct on cellulosic fibres, but these are converted to blacks of excellent fastness to light by aftertreating in hypochlorite solutions. It is recommended for use on curtaining, furnishings, and dress linens, but not where guaranteed fastness to washing and boiling is demanded. The light fastness of dyeings is practically unaffected by crease-resist finishing. Fastness figures include— Light (daylight SNV) 6–7, washing (100°C.) 4, chlorination (2 g. active chlorine per litre) 5.

SANDOTHRENE OLIVE F-N2B— This vat dye gives greens of very good fastness to severe washing and bleaching, and is particularly recommended for curtainings and furnishings and for dark greens on awnings. The light fastness of dyeings is completely unaffected by crease-resist finishing. Fastness figures on cotton include— Light (daylight SNV) 8, washing (100°C.) 5, chlorine (2 g. active chlorine per litre) 4–5.

CUPROFIX YELLOW C-2RLN— This direct dye gives bright reddish yellows of excellent fastness to light and very good fastness to washing on all types of cellulosic fibres when aftertreated on the fibre with either copper sulphate and acetic acid, Cuprofix SL, or Resofix CU. Dyeings are only very slightly affected in hue and light fastness by crease-resist finishing. It is suitable for use in combination with all other Cuprofix C dyes. Fastness figures for a dyeing on cotton aftertreated with copper sulphate–acetic acid include— Light (daylight SNV) 6–7, washing (60°C. SNV) 4–5, perspiration (SNV) 4–5.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I— PLANT; MACHINERY; BUILDINGS

Engraving of Printing Rollers

H. G. Jakob and G. Brink

Melliand Textilber., **39**, 1371–1376 (Dec. 1958)

All stages of the engraving of machine printing rollers by conventional and automatic pantograph methods (economical for large patterns) and by steel rolls (giving high resolution) are discussed thoroughly. 17 illustrations of machines and processes are provided. S.M.J.

Chromium and Copper Plating of Printing Rollers

H. G. Jakob and V. Schmudlach

Melliand Textilber., **40**, 77–83 (Jan. 1959)

An exhaustive review of practical procedure. Under chromium plating the authors discuss the electrolytic processes, the sequence of operations, the relationship between liquor composition, temp., and current density, and testing methods. They then describe pretreatment of rollers and copper deposition, internally, externally, and on steel rollers. Finally, detoxification of CrO_3- and cyanide-bearing effluents is discussed. 6 illustrations. S.M.J.

Current Applications of Infrared Heating

P. Rochas *Teintex*, **24**, 9–28 (Jan. 1959)

The setting of man-made fibres, the Thermosol process for polyester fibres, the continuous dyeing of nylon with reactive dyes, and the application of resin–pigment systems are discussed. S.R.C.

PATENTS

Treating Textiles with Liquids

Dexdale Hosiery Mills *USP* 2,845,787

A package dyeing machine is provided with a valve and a simplified circulating system wherein a separate side opening to the vessel is eliminated. This results in more uniform and evenly distributed flow of liquor through the goods. C.O.C.

Drying Yarn Packages or the like

Linen Thread Co. *BP* 807,794

The packages are mounted on spindles in a truck and are then taken to a source of hot air. The truck is so designed that it can be coupled to the hot air supply so that the hot air flows into the spindles and so through the packages to dry them. This saves much handling of the packages. C.O.C.

Eliminating Trapped Air from Yarn impregnated with Liquid Resin
Specialities Development Corpn. *USP* 2,848,354

Waxing of Yarn
Thomas Holt *BP* 808,067
When yarn is being waxed in a winding machine by passing it between a disc of wax mounted on a rotating spindle and the surface of the base on which the spindle is mounted, thorough waxing of the yarn irrespective of the speed of the yarn and equal wearing of the surface of the wax disc are obtained by slowing down the rotation of the waxing device relative to the speed of the yarn. This is done by means of an escapement movement incorporated in the waxing device which only permits intermittent rotation of the wax disc at a predetermined rate. In addition there is the advantage that if the yarn should break the pull of the yarn is insufficient to overcome the inertia of the waxing device and so the tendency of the yarn end to wrap around the spindle is eliminated. C.O.C.

Warp Sizing
A. C. Adams *USP* 2,849,784
The rear sizing roller and its associated impregnating roller are mounted within the vat with their axes parallel to and below the surface of the size so that yarn entering the vat is carried over the rear size roll, then downwards to a nip below the surface of the size, then around the underside of the impregnating roll. From there it passes upwardly over the second partly immersed size roll and finally between a nip above the surface of the size and formed by the second size roll and or finishing roll mounted vertically above it. C.O.C.

Continuous Wet Treatment of Fabrics
A. Libbrecht and P. Libbrecht *BP* 809,199
Three driving rollers are arranged successively in the same horizontal plane; the axis of the centre one is fixed, whereas the axes of the others are movable with respect to it so as to regulate the nip between them and the axially stationary roller. Fabric passes down between the first nip rollers, through an impregnating bath and then up through the second nip. There may be a bath of impregnating liquor held between the rollers forming the first nip. C.O.C.

Heating Cloth by Direct Contact with Steam
DuP *BP* 807,629
Moist, impregnated cloth is continuously fed into the enclosed upper part of a J-box and continuously piled in the J-box below the upper part so that it forms a seal. The piled fabric is not allowed to enter into the upper part of the J-box. Steam is continuously supplied to the upper part of the J-box, preferably being introduced adjacent to the seal of piled fabric, and flows countercurrent to the fabric and in direct contact with it. C.O.C.

Treating Cloth in Folds
R. Holzhauer *BP* 808,195
The fabric passes through a chamber, e.g. for steaming during bleaching, on a series of endless conveyors. It is laid in folds and the positions of the various conveyors and their speeds are such that after being laid flat on the first conveyor the folds are turned upright in which position they pass through the greater length of the chamber, being returned to the horizontal position as they reach the exit of the chamber. The machine is very compact and efficient. C.O.C.

Steam-heated Drying Cylinder
Armstrong Machine Works *BP* 808,303
A simple syphon-type device for draining condensate from the cylinder whatever its speed of rotation. C.O.C.

Shrinking Knitted Fabrics
K. I. J. Rosén *USP* 2,849,782
Apparatus in which knitted fabric is carried between two longitudinally moving webs of endless stainless steel helical springs extending in the direction of the fabric's passage. These helical springs are progressively contracted in their direction of movement and so exercise a compressive shrinkage action on the fabric. The whole is enclosed in a chamber so that the fabric can be steamed during the treatment.

S. G. Andersson *USP* 2,849,783
The springs are heated electrically. C.O.C.

Silk Screen Printing Machine
American Automation *USP* 2,846,946
A machine in which the several colours are printed on articles, e.g. shirts, in continuous succession if necessary, allowing time between each printing for the one colour to dry before the next colour is printed. The articles may be fed to the machine either singly or in groups. C.O.C.

Textile Folding or Rigging Machine
William Whiteley & Sons *BP* 808,699
A machine which automatically corrects inaccuracies in the rigging to ensure that the selvedges lie against one another.

BP 808,700
A machine which ensures that the weft is at right angles to the selvedges. C.O.C.

Metal-coated Glass Fibres
Owens-Corning Fiberglas Corpn. *USP* 2,849,338
Apparatus is described for the coating with metal of glass fibres containing an oxide of another metal in addition to oxides of Si, Al, and Na. C.O.C.

Machines for Applying Leather Finishes
United Shoe Machinery Corpn. *USP* 2,848,974
A machine which automatically coats or seasons leather at a rapid rate. C.O.C.

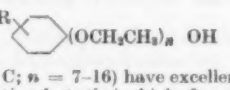
III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Sugar Detergents
N. Pilpel *Research*, **12**, 68–74 (Feb. 1959)
A new class of synthetic detergent, based on sucrose esters, is now commercially available. Their development is described and their chemical and physical properties compared with those of alkylaryl sulphonates and a nonionic surface active agent produced by condensing tall oil with polyoxyethylene. Their introduction may help to solve certain difficulties which have occurred in the treatment of sewage. 27 references. C.O.C.

Non-ionic Emulsion of Olive Oil
L. Mauri *Investigación e Información Textil*, **1**, 270–276 (Oct.–Dec. 1958)
With appropriate HLB (hydrophile–lipophile balance) $\geq$ 5% of emulsifying agent is necessary. Increase in the acid value requires increased HLB. Cetyl alcohol additions have no synergistic effect. S.R.C.

PATENTS

Low-foaming Non-ionic Surface-active Agents
Rohm & Haas Co. *USP* 2,850,535
Compounds of formula—



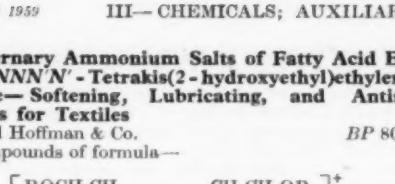
(R = Alk of 8–9 C; n = 7–16) have excellent wetting and detergent properties but their high foaming properties often adversely affect their use as detergents. This foaming property is much reduced without diminishing their detergent or wetting properties by heating them at 160–250°C. with an acid-treated clay or a strongly acidic cation-exchange resin. C.O.C.

Antistatic Finish Fast to Washing
American Cyanamid Co. *USP* 2,848,413
The mixture of the polymethylol derivatives of a polyethoxamer of *NNN*-tris-(2-hydroxyethyl)-*N*-(4-amino-6-anilino-*s*-triazinylmethyl)ammonium chloride and *N*-(4-amino-6-anilino-*s*-triazinylmethyl)-*NNN*-trimethylammonium chloride obtained by reacting a chloroacetoguanamine with triethylamine, HCHO and an ethoxamer of tris-(2-hydroxyethyl)amine impart durable wash-fast antistatic properties to nylon, polyester fibres, polyacrylic fibres, and other fibres. C.O.C.

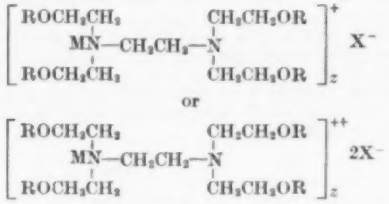
Carboxymethyldextran as a Soil-suspending Agent for Incorporation in Detergents
Commonwealth Engineering Co. *USP* 2,847,385
Carboxymethyldextran is a very effective soil-suspending agent for combination with detergents. C.O.C.

Quaternary Ammonium Salts of Fatty Acid Esters of *NNN'N'*-Tetrakis(2-hydroxyethyl)ethylenediamine— Softening, Lubricating, and Antistatic Agents for Textiles
Arnold Hoffman & Co. *BP* 808,265

Compounds of formula—



or



(R = H or aliphatic acyl of 12–18 C, at least one R being acyl; M = univalent hydrocarbon radical of 1–4 C; X = anion, z = valency of X), e.g. *NNN'N'*-tetrakis(2-hydroxyethyl)-ethylenediamine mono-, di-, tri- or tetra-stearate, are excellent softeners and lubricants for textiles. They have unusual antistatic properties and are resistant to heat. They are especially suitable for use on acetate rayon, nylon, and vinyl resins. C.O.C.

Imparting Good Foaming Power below 100°F. to Sulphated and Sulphonated Anionic Detergents
Thomas Hedley & Co. *BP* 809,060

Addition of 5–60% by weight of an amide of formula

$R^1CO{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}N(CH_2CH_2OH)_2$

or

$R^1CO{\cdot}NCH_3{\cdot}CH_2(CHOH)_4CH_2OH$

or

$R^2{\cdot}NHCO(CHOH)_4CH_2OH$

(R^1CO = acyl radical of a fatty acid of 10–14 C; R^2 = Alk of 10–14 C) to sulphated or sulphonated anionic detergents gives them good foaming power at $< 100°F$. C.O.C.

Lubricants for Glass Fibres
Arthur D. Little *USP* 2,854,356

Organic nitrogenous fluosilicates, e.g. tallow amine fluosilicate, are useful lubricants for glass fibres during processing. C.O.C.

β-Amino Lower Alkyl Carbamate–Formaldehyde Adducts for Improving the Recovery from Creasing and Dimensional Stability of Cellulosic Textiles
Dow Chemical Co. *USP* 2,850,407

The adducts of 2–4 moles of HCHO with 1 mole of the product obtained by treating 1 mole of urea with 0·7–1·5 moles of an alkylene oxide and having the formula $H_2N{\cdot}CHR^1{\cdot}CHR^2{\cdot}O{\cdot}CO{\cdot}NH_2$ (R^1 and R^2 = H or Alk of 1–4 C, $R^1 + R^2 = < 5$) are used as resin finishes for textiles. C.O.C.

β-Amino Lower Alkyl Carbamate–Epichlorohydrin Adducts for Improving the Recovery from Creasing and Dimensional Stability of Cellulosic Textiles
Dow Chemical Co. *USP* 2,850,408

The products obtained by treating compounds of formula $H_2N{\cdot}CHR^1{\cdot}CHR^2{\cdot}O{\cdot}CO{\cdot}NH_2$ (see previous abstract) when treated with epichlorohydrin yield self-curing aqueous solutions at pH 6–7 useful as resin finishes for textiles. C.O.C.

Permanent Waving Composition
Gillette Co. *BP* 809,264
USP 2,847,351

A highly effective composition which works in cold solution consists of a mixture of (1) a water-soluble disulphide of thioglycerol or of thioglycollic acid or a water soluble salt thereof and (2) an odourless reducing agent which reduces the disulphide to the corresponding mercaptan when the mixture is dissolved in water. Some of these reducing agents, e.g. thiomalic acid or thiourea dioxide (C.I. Reducing Agent 11) are comparatively ineffective hair waving agents by themselves even when used in large amounts, while in the case of others, e.g. dimercapto adipic acid, which are effective hair waving agents when used in sufficient quantity, they are much less effective than the above mixtures used at the same strength and pH. C.O.C.

Size for Nylon
Monsanto *USP* 2,854,357

The water-soluble free acid form of an olefin–maleic interpolymer having 0–100% of its maleic units monoesterified with an alkanol is a non-tacky size for nylon irrespective of atmospheric humidity. The olefin is of 2–3 C and the interpolymer has specific viscosity of $< 0{\cdot}1$ as a 1% by weight solution in dimethyl formamide at 25°c. C.O.C.

Stannous Octoate, Oleate, and Naphthenate as Catalysts for Silicone Water-repellent Agents
General Electric Co. *USP* 2,854,424

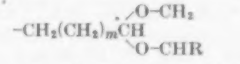
Mixtures of methyl polysiloxanes containing only a small amount of methyl hydrogen polysiloxane can be used to give a water-repellent finish having a high initial spray rating and good durability to dry cleaning, if stannous octoate, oleate or naphthenate is used as the catalyst. C.O.C.

Carbohydrate Derivatives— Plasticisers, Water-repellent Agents, etc.
General Aniline *USP* 2,853,485

Carbohydrates when dissolved in 2-pyrrolidone or *N*-methyl-2-pyrrolidone react readily with various reagents to yield products having a wide variety of uses. Thus octastearyl sucrose is useful as a water-repellent agent for cotton and sucrose tetra-acetate as an adhesive and plasticiser. C.O.C.

Organosilicon Dioxolanes— Water-repellent Agents
Dow Corning Corpn. *USP* 2,846,448

Monomeric organosilicon compounds in which at least some of the Si atoms are attached to a radical of formula—



(m = 1–16; R = Alk of 1–3 C) and which also contain hydrolysable substituents, are used for rendering textiles, paper, etc. water-repellent. C.O.C.

Water-repellent Agents
Boehme Fettchemie *BP* 807,904

Treating solutions of basic polyvalent metal salts (e.g. of Al or Cr) in alcohols of < 5 C with acid phosphoric acid esters of hydroxylic compounds of > 7 C yields solutions of organic metal complex compounds. These solutions can be diluted with water to any desired extent and used as impregnants for imparting water-repellency to textiles, skins, feathers, and paper or for rendering plastics, porous ceramics, and metal surfaces hydrophobic and also for making paints water-repellent. C.O.C.

Bromine-containing Nitrile Methylolphosphorus Polymers as Flame-resisting Agents
Albright & Wilson *BP* 807,236

The product obtained by reacting together (a) tris-(hydroxymethyl) phosphine oxide or a tetrakis(hydroxymethyl) phosphonium salt or polymethylol derivatives thereof containing > 1 methylol group directly linked to P, (b) a compound of mol.wt. > 800 containing trivalent N and several H atoms and/or methylol groups and/or terminally unsaturated alkenyl radicals, each attached to trivalent N, and (c) a polyfunctional compound containing Br attached to C and capable of condensing with HCHO and/or esterifying methanol, are better flame-resisting agents than those described in *BP* 740,269, 761,985, and 764,314 (J.S.D.C., **72**, 40 (1956); **73**, 61, 216 (1957)), which are similar in constitution but contain no Br. The proportion of the reactants is such that they contain $< 1\%$ P (in (a)) and for every part of this P 0·5–20·0 parts of nitrilo N in (b) and 9·3–15·0 parts of Br in (c). C.O.C.

Synergistic Fungicidal Mixtures of Chlorophenols and Pyrones for treating Vegetable Fibres
Polaquimia *USP* 2,849,360

A mixture of an Na or K salt of 2,4,5-trichlorophenol, 4-chloro-2-phenylphenol, 6-chloro-2-phenylphenol, 2,3,4,6-tetrachlorophenol or pentachlorophenol and 3-acetyl-6-methyl-pyrandione-2,4 or other pyrone compound described in *USP* 2,229,204 and 2,653,943–5, is an excellent preservative for hard vegetable fibres, e.g. henequen fibre. The rotproof given is resistant to leaching. C.O.C.

Drier Accelerators
Harshaw Chemical Co. and Carlisle Chemical Works
USP 2,852,405

Organic compounds in which trivalent N is conjugated with a resonating system containing an oxygen containing group (e.g. OH, alkoxy or COOH), e.g. picolinic acid, are excellent accelerators for primary driers and enable considerable reduction in the amount of metal drier required in drying oil compositions. C.O.C.

Aluminium Alkoxide Derivatives as Coupling, Gelling, Thickening, Waterproofing, and Drying Agents
Hardman & Holden *BP* 809,309

1 mol. of Al alcoholate or phenolate is heated with 1 mol. of a monocarboxylic acid or anhydride so that 1 mol. of alcohol or phenol distils off. The product is then heated in presence of water until another mol. of alcohol or phenol has distilled off and a hydroxy-alkoxy- or hydroxy-aryloxy-aluminium acylate is formed. Depending upon (a) type of carboxylic acid used, (b) end groups, and (c) degree of polymerisation the products can be viscous liquids, resins, waxes or rubbery materials. They find use in coatings, plastics, etc. C.O.C.

Purification of Precipitated Silica (IV p. 227)
Titanium Ester Complexes (V p. 228)
Size for Nylon (X p. 234)
Fungicides—Rot and Water Repellent Finish (X p. 234)
Polyurethanes for Coating Cloth and for producing Fibres and Films (XIII p. 236)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

The Colour Committee of the Deutsche Forschungsgemeinschaft. List of Pigments and Dyes for Cosmetics
S. W. Sovci
Z. Lebensm.-Untersuch. u. -Forsch., **108**, 189–195 (1958)

Toxicological Data on Dyes and their Suitability for Foods in Various Countries *Ibid.*, 195–8

Relationship between the Absorption Spectrum of Oxidation-Reduction Dyes and pH
Y. Koyama, M. Masuda, and M. Yoshikawa
Osaka Daigaku Igaku Zasshi, **10**, 1193–5 (1958): *Chem. Abs.*, **53**, 720 (10 Jan. 1959)

In the visible absorption spectrum of aqueous solutions of some oxidation–reduction dyes at pH 3·2–12·0 the absorption maxima were (mμ.): Methylene Blue (C.I. Basic Blue 9) 665, Phenosaframine 540, Nile Blue (C.I. Basic Blue 12) 640, Thionine (C.I. Basic Blue 25) 600, Crystal Violet (C.I. Basic Violet 3) 590, indigo disulphonate (C.I. 73015) 615, indigo trisulphonate 595. The molecular extinction coefficient, E, at the absorption maximum decreased as the pH of the medium increased and dE/d pH was approx. $-0{\cdot}02$ at pH 3·2–10·0 for all the dyes; at pH $> 10{\cdot}0$ E decreased abruptly with little increase in pH. On the other hand, the position of the maximum absorption was scarcely influenced by change in pH. C.O.C.

Preparation of Merocyanine Dyes
J. Świderski and J. Oszczapowicz
Roczniki Chem., **32**, 413–4 (1958): *Chem. Abs.*, **53**, 339 (10 Jan. 1959)

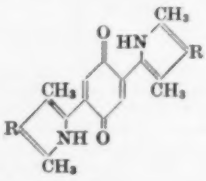
Merocyanine dyes may be simply prepared from 2 - thiono - 3 - ethyl - 4 - oxo - 5 - (5 - anilino - 2,4 - pentadienylidene)thiazolidine by heating with an amine in alcohol. When piperidine is used the 5′-piperidino analogue is produced. C.O.C.

Polymeric Phthalocyanines—II
C. S. Marvel and M. M. Martin
J. Amer. Chem. Soc., **80**, 6600–6604 (20 Dec. 1958)

Co-condensation of 3,3′,4,4′-tetracyanodiphenyl ether, phthalonitrile and copper-bronze under phthalocyanine-forming conditions results in formation of dimeric or trimeric products. No high weight molecular polymeric phthalocyanines are formed. A high molecular weight cross linked polymer of 3,3′,4,4′-tetracyanodiphenyl ether can be prepared by fusion with copper-bronze. Similar results are obtained in the cocondensation in presence of sodium amylate. All the polymers decompose in air at 350°C. C.O.C.

Addition of Pyrroles to 1,4-Benzoquinone
E. Bullock *Canadian J. Chem.*, **36**, 1744 (Dec. 1958)

The formation of coloured adducts from quinones and 1,2-pyrroles involves the loss of four hydrogens between 1 mol. of the former and 2 mol. of the latter, quinol being a by-product. The infrared spectra of the benzoquinone adducts of 2,4-dimethyl-(I*a*) and cryptopyrrole (I*b*) indicate a "bonded" NH band at 3360 cm.$^{-1}$, so that formula I*a* and I*b* give a satisfactory explanation of the intense colour of the cpd. and their behaviour with acid, viz. the violet of I*a* and the blue of I*b* in *NN*-dimethylformamide, turning with conc. HCl to yellow and very pale green respectively, with restoration of the violet and blue on neutralisation.



(I*a*: R = H; I*b*: R = C_2H_5) H.H.H.

Animal Tests in the Chronic Compatibility of β-Carotene (C.I. 75130), Lycopene (C.I. 75125), 7,7′-Dihydro-β-carotene, and Bixin (C.I. 75120)
G. Zbinden and A. Studer
Z. Lebensm.-Untersuch. u. -Forsch., **108**, 111–134 (1958): *Chem. Abs.*, **53**, 594 (10 Jan. 1959)

Tests on rats, rabbits, and dogs show that the above four carotinoid compounds are non toxic and can be used without fear as food dyes. C.O.C.

Colouring Matters of Australian Plants. VI—Hemocorin: Structure of the Aglycon
R. G. Cooke, B. L. Johnson, and W. Segal
Australian J. Chem., **11**, 230–5 (1958): *Chem. Abs.*, **53**, 311 (10 Jan. 1959)

Evidence which suggests that the aglycon of hemocorin is the potentially tautomeric 2,6-dihydroxy-5-methoxy-9-phenylperinaphthen-1-one. C.O.C.

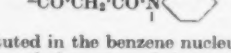
Mica (C.I. Pigment White 20) in Paints
V. Charrin *Peintures, Pigments, Vernis*, **34**, 415–6 (1958): *Chem. Abs.*, **53**, 733 (10 Jan. 1959)

Various lepidolith deposits in France yield products suitable as paint pigments. C.O.C.

PATENTS

Colour Couplers
Gevaert *BP* 808,276

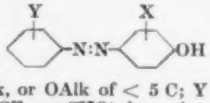
Yellow photographic images are obtained by use of a primary amino aromatic developing agent in presence of an aroyl acetarylide whose



group is substituted in the benzene nucleus by SO_3H, the H atoms of the methylene group being optionally replaced by substituents which readily split off on colour development. These couplers are made by condensing an aroylacetic ester with a compound of formula $NH_2{\cdot}Ar{\cdot}SO_2F$ and then hydrolysing the resulting sulphofluoride into the corresponding sulphonic acid. C.O.C.

4-Hydroxyazobenzenes—Yellow Disperse Dyes for Polyester Fibres
FH *BP* 808,174

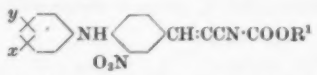
4-Hydroxyazobenzene and its derivatives—



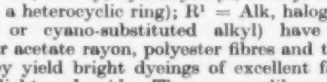
(X = H, Cl, Alk, or OAlk of < 5 C; Y = H, Cl, Alk, or OAlk of < 5 C, CF_3, or CHO) dye polyester fibres yellows of good light fastness. Thus the monoazo compound *m*-chloroaniline→*o*-cresol dyes polyethylene terephthalate reddish yellow. E.S.

Nitro Diarylamine Methin Disperse Dyes
General Aniline *USP* 2,849,447

Dyes of formula—



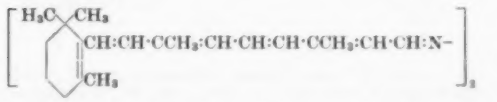
(x and y = H, NO_2, Alk, alkoxy, Hal or $SO_2NR^2R^3$(R^2 and R^3 = H, Alk, alkoxy, hydroxyalkyl, or acyl or together may form a heterocyclic ring); R^1 = Alk, halogeno alkyl, hydroxy- or cyano-substituted alkyl) have excellent affinity for acetate rayon, polyester fibres and the like on which they yield bright dyeings of excellent fastness to washing, light and acids. They are readily prepared by condensing 4-chloro-3-nitrobenzaldehyde with a cyanoacetic ester and further condensing the product with monocyclic, carbocyclic, primary aromatic amines. Thus ethyl α-cyano-3-nitro-4-chloro cinnamate condensed with aniline yields the greenish yellow disperse dye—



C.O.C.

Yellow Dye for Edible Fats
Eastman Kodak Co. *BP* 807,865

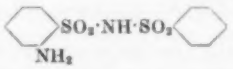
The compound—



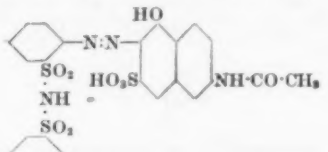
when added to margarine colours is yellow and also provides vitamin A fortification. It is produced by treating vitamin A aldehyde with hydrazine or hydrazine-hydrate. C.O.C.

Orange and Red Monoazo Acid Dyes
FBy *BP* 806,938

N^1-Benzenesulphonyl derivatives of orthanilamide—



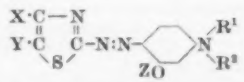
(the benzene rings may contain non-solubilising substituents) are diazotised and coupled with naphthol- or aminonaphthol-monosulphonic acids to give orange and red dyes applicable to wool, nylon, etc. from weakly acid baths. Thus o-nitrobenzenesulphonyl chloride was condensed with benzenesulphonamide, and the product reduced in presence of Raney nickel, diazotised and coupled in presence of Na_2CO_3 with *N*-acetyl-J acid, giving—



which dyes wool yellowish orange. E.S.

Metallisable Monoazo Disperse Dyes
Eastman Kodak Co. *USP* 2,835,661

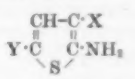
Monoazo dyes—



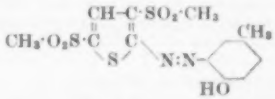
(X, Y = H, CF_3, CCl_3, NO_2, or Alk; Z = H or Alk; R^1,R^2 = H, Alk, or hydroxyalkyl), may be metallised on cellulose acetate, esp. with $Ni(SCN)_2$, or their metal-complexes may be incorporated in the spinning solutions. Thus the monoazo compound 2-aminothiazole→*NN*-diethyl-*m*-aminophenol dyes cellulose acetate orange, converted into deep violet fast to light and washing by padding with aq. 2% $Ni(SCN)_2$ and steaming for 10–20 min. at 5 lb. per sq.in. pressure. E.S.

Yellow and Orange Monoazo Disperse Dyes from 2-Aminothiophene Derivatives
Eastman Kodak Co. *USP* 2,825,726

2-Aminothiophene derivatives—



(X = NO_2 or SO_2·Alk; Y = SO_2·Alk or SO_2·NHAlk) are diazotised and coupled with mono- or di-hydric phenols or with 5-methyl (or 5,5-dimethyl)*cyclo*hexane-1,3-dione to give the title dyes. Thus 2-acetamido-3,5-bis(chlorosulphonyl)thiophene (*J. Amer. Chem. Soc.*, **73**, 615 (1951)) is reduced with Na_2SO_3 in presence of aq. NaOH to give the disulphinic acid, which is reacted with sodium chloroacetate to give the bis(methylsulphonyl) compound (I), or with ethyl iodide to give the bis(ethylsulphonyl) compound. Hydrolysis of I with hot aq. HCl then gives 2-amino-3,5-bis(methylsulphonyl)thiophene, which when diazotised in nitrosylsulphuric acid in presence of acetic-propionic acid mixture at 0° to −5°c. and coupled with *p*-cresol gives—

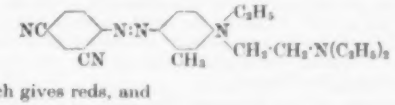


which dyes cellulose acetate, nylon, polyester, sulphone polyester, and acrylonitrile graft polymer fibres orange. E.S.

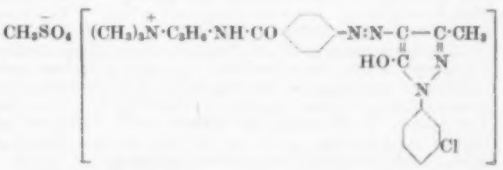
Monoazo Dyes for Polyacrylonitrile Fibres
FBy *BP* 808,713

Monoazo dyes free from SO_3H and COOH groups and having amino or quaternary ammonium groups not directly attached to the aromatic nuclei have good affinity for polyacrylonitrile fibres.

Examples are—



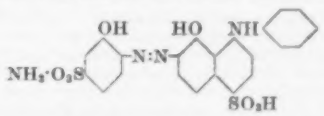
which gives reds, and



a greenish yellow. E.S.

Blue–Green Afterchrome Monoazo Dyes
Gy *BP* 808,919

2-Amino-phenols having in the 5-position a group SO_2·R (R = NH_2, subst. amino, Ar, Alk, or aralkyl), and optionally substituted in the 4-position with e.g. Hal or CH_3, are diazotised and coupled with 1-phenylamino-8-naphthol-4-sulphonic acid (the phenyl group may contain an Alk or OAlk group) to give the title dyes. Thus 2-aminophenol-5-sulphonamide is diazotised and coupled with 1-anilino-8-naphthol-4-sulphonic acid to give—



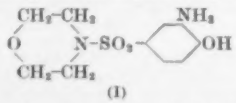
E.S.

Yellow Monoazo Disperse Dyes
S *BP* 807,696

N-Benzoyl (or toluyl)-*p*-phenylenediamine is diazotised and coupled with *o*-coupling phenols to give disperse yellows of good fastness properties. Thus the monoazo dye *N*-benzoyl-*p*-phenylenediamine→*p*-cresol is faster to washing and sublimation on cellulose acetate, nylon, and Terylene than *N*-acetyl-*p*-phenylenediamine→*p*-cresol (C.I. Disperse Yellow 3). E.S.

Yellow, Orange, and Brown Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, etc., having a Morpholine Residue
General Aniline *USP* 2,841,577

The Co complexes of monoazo compounds from diazotised 2-aminophenol-4-sulphonmorpholide—



(I)

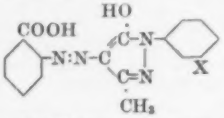
coupled with acetoacetanilide and its derivatives, dye wool and nylon yellow, faster to washing than the similar dyes having a sulphonamide group instead of the sulphonmorpholide group. Thus I is diazotised and coupled with acetoacet-2,4-xylidide, and the product refluxed in isopropanol with $CoCl_2$. The Co complex, after mixing with sodium naphthalene-β-sulphonate and drying, dyes wool and nylon from a dispersion containing ammonium sulphate.

USP 2,834,772

The same diazo component is coupled with 3-methyl-1-phenyl-5-pyrazolones, to give monoazo compounds whose Co and Cr complexes dye wool and nylon orange to yellow-brown from aqueous dispersion, of good wet fastness. Thus the Cr-complex of I → 1-(2′,5′-dichlorophenyl)-3-methyl-5-pyrazolone is an orange. E.S.

Yellow Monoazo Pyrazolone Chromium Complexes for the Mass Coloration of Synthetic Fibres
S *BP* 807,695

The title products contain 1 atom of Cr to 2 mol. of monoazo compounds—



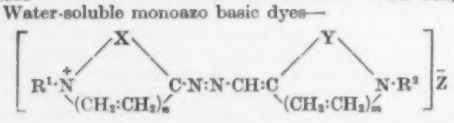
(X = non-water-solubilising substituent, e.g. Cl, Br, Alk, OAlk, NH_2 or CN). Thus the monoazo compound in which X = Cl is heated with chrome alum in formamide at 130°c., and the resulting ammonium salt of the Cr-complex converted into the Na salt. E.S.

Stabilisation of the Copper Complex of C.I. Direct Blue I
American Cyanamid Co. *USP* 2,834,773

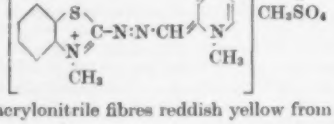
The copper complex of the disazo compound dianisidine ⇉(Chicago acid)$_2$, described in *USP* 2,036,159, has poor stability to storage, and loses ca. 30% of its strength and gives redder and duller dyeings after storing for 6 months. If, however, it is treated or mixed with 1–10% of an organic sequestering agent, e.g. sodium ethylenediaminetetraacetate, its stability is greatly increased. E.S.

Monoazo Basic Dyes for Polyacrylonitrile Fibres
BASF *BP* 808,308

Water-soluble monoazo basic dyes—



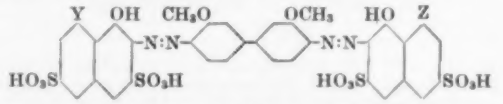
(*n*, *m* = 0 or 1; X, Y = divalent atoms or radicals which make up the nitrogenous rings to 5- or 6-membered rings; R^1, R^2 = Alk, Ar, aralkyl, or cycloalkyl, and R^2 may = H; $\bar{Z}$ = inorganic, organic, or complex anion) give strong, bright dyeings on polyacrylonitrile and its copolymers. They are prepared by coupling suitable diazotised heterocyclic amines with methylene bases, or by condensing the corresponding hydrazones with the appropriate aldehydes, followed if desired by treatment with quaternising agents such as CH_3I, dimethyl sulphate, etc. Thus the compound—



dyes polyacrylonitrile fibres reddish yellow from an acetic acid bath. E.S.

Blue Metal(Copper)-complex Disazo Direct Dyes
Ciba *BP* 808,063

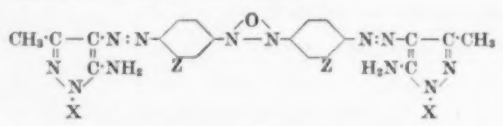
Copper-complexes obtained by coppering with demethylation of OCH_3 groups of disazo compounds—



(Y = OAlk of < 5 C containing an OH or OAlk group; Z = Y, unsubst. OAlk, or OH) dye cellulose blues of good brightness. Thus tetrazotised *o*-dianisidine is coupled with 2 mol. of 1-β-hydroxyethoxy-8-naphthol-3,6-disulphonic acid (from chromotropic acid and ethylene chlorhydrin) in presence of Na_2CO_3 and pyridine or picoline. Refluxing with ammoniacal $CuSO_4$ in presence of monoethanolamine gives the copper-complex, which dyes cotton bright greenish blue. E.S.

Red Disazo Direct Dyes for Aftercoppering having an Azoxy Group
FBy *BP* 808,798

Disazo dyes of general formula—



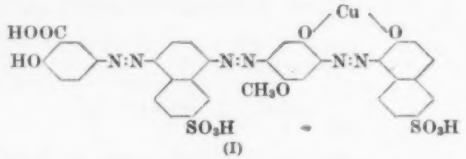
(X = aryl; Z = metal-complex forming group) give fast, dischargeable reds on cellulose by an aftercoppering process. Thus diazotised 5-nitroanthranilic acid is coupled in presence of acetic acid with 3-methyl-1-phenyl-5-aminopyrazole, and the nitromonoazo compound so formed is reduced at 50–60°c. with glucose in presence of aq. NaOH to give the azoxy compound (X = phenyl; Z = COOH). E.S.

Azo Solvent Dyes from Alkyl-β-naphthols
American Cyanamid Co. *USP* 2,850,493

Alkyl derivatives of β-naphthol, in which the alkyl group has > 3 C atoms, when coupled with suitable diazo- and tetrazo-compounds, give azo dyes of deep colour and good solubility in hydrocarbons. Thus the monoazo compound *o*-toluidine→amyl-β-naphthol is an orange red, and the disazo compound dianisidine⇉(diamyl-β-naphthol)$_2$ is a red violet. E.S.

Grey Metallisable Trisazo Direct Dyes
Ciba *BP* 807,575

Copper complexes of the type—



(I)

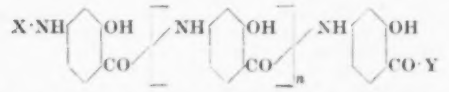
and the corresponding *oo′*-dihydroxyazo compounds formed by removal of the Cu atom, dye cellulose grey of good wet fastness by an aftercoppering process. They are prepared by coppering (with dealkylation) the *o*-alkoxy-*o′*-hydroxyazo compounds. Thus the *o*-methoxy-*o′*-hydroxytrisazo compound 5-aminosalicylic acid→1-naphthylamine-7-sulphonic acid→2,5-dimethoxyaniline →2-naphthol-6-sulphonic acid is stirred at 95°c. for 5 hr. in aq. ammoniacal $CuSO_4$ in presence of pyridine. Treatment with ca. 1% aq. HCl removes unwanted Cu from the salicylic acid residue, giving I. E.S.

Readily Diazotisable Dispersions of Arylamines
General Aniline *USP* 2,845,326

Mixtures of 10–30% of arylamines such as *o*-aminoazotoluene with sodium nitrite, 10–25% of a non-ionic surface active agent, 2–10% of a cationic surface active agent, 25–40% of an organic solvent (e.g. ethylene glycol ethyl ether or dimethylformamide) and 10–20% of water, give stable dispersions which with dil. HCl readily give diazo solutions suitable e.g. for the development of azoic dyeings. E.S.

Condensation Products of p-Aminosalicylic Acid— Coupling Components for Azoic Browns
American Cyanamid Co. *USP* 2,841,575

Coupling components—

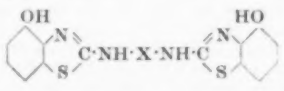


(X = H or acyl; Y = OH or NH Aryl; n = 0 or integer) give azoic brown with many common diazo components. They may be made by heating *p*-aminosalicylic acid with thionyl chloride, followed if desired by blocking of the terminal NH_2 and COOH groups by reacting with an acylating agent and/or an arylamine. Thus *p*-aminosalicylic acid is stirred in dioxane at 71–75°C. with thionyl chloride to give a product (I) of mol.wt. ca. 480; a similar reaction carried out in presence of pyridine gives a product of mol.wt. ca. 700. On cotton, I couples with diazotised *m*-chloroaniline or dianisidine to give red-browns.

E.S.

Bis(benzthiazole) Compounds— Coupling Components for Azo Dyes
ICI *BP* 808,191

Two mol. of 2-amino-4-hydroxybenzthiazoles are condensed with 1 mol. of phosgene, cyanuric chloride, etc. to give compounds—

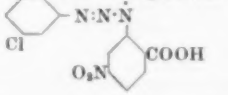


(X = CO, terephthaloyl, fumaroyl, or 2,4-divalent-1,3,5-triazinyl which may be subst. in the 6-position by Cl, OH, NH_2, or subst. amino; the benzthiazole nuclei may contain Alk, OAlk, Hal, or SO_3H groups) which are coupling components for azo dyes. Thus 2-amino-4-hydroxybenzthiazole is dissolved in aq. NaOH, and phosgene is passed in at < 30°C., finally heating to 40–45°C. to give the product in which X = CO.

E.S.

Azoic Dyes developable by Neutral Steaming
FBy *BP* 808,262

The triazenes obtained by condensing *N*-(2-carboxy-5-nitrophenyl)glycine (I) with diazotised weakly negatively substituted anilines (e.g. halogeno- and dihalogeno-anilines and -toluidines, alkoxy- and trifluoromethyl-anilines, dianisidines, and anilinesulphonamides), although stable cold regenerate the diazo compound readily on steaming. Hence, by mixing them with suitable coupling components, azoic dyeings and prints can be produced by steaming under neutral conditions. Thus diazotised 2-amino-4-chlorotoluene is slowly run into a solution of I in aq. Na_2CO_3 to give the triazene—



which is mixed with a solution in alcoholic NaOH of 3-hydroxy-2-naphtho-*o*-phenetidide suitably thickened, printed on cotton fabric and dried. Steaming for 5–10 minutes under neutral conditions develops the bright scarlet azoic dye.

E.S.

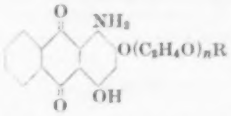
Violanthrone Vat Black
General Aniline *BP* 807,595

A black vat dye of high light fastness is

cf. *USP* 2,819,270 (J.S.D.C, **74**, 876 (1958)). E.T.

Anthraquinone Disperse Dyes
S *BP* 807,591

Brilliant, level, fast-to-light reds on cellulose esters or ethers, polyamides, polyacrylonitriles or aromatic polyesters are obtained with disperse dyes of formula—

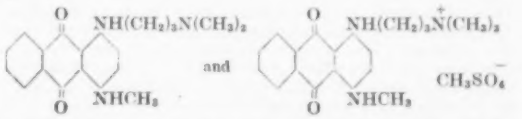


(R = Alk of 1–6 C; n = 2 or 3). The dyes are made by reacting an alkali-metal salt of 1-amino-4-hydroxyanthraquinone-2-sulphonic acid (1 mol.) with a monoalkyl di- or tri-ethylene glycol ether (> 1 mol.) at 100–200°C. Excess of glycol ether is preferred so that it acts as reaction component and solvent. Water-free conditions preclude undesirable by-products; thus, KOH is dissolved in the glycol ether and the water formed is removed by a current of dry air at 120°C. before reaction. The dye is separated by neutralising with acid, removing the solvent, filtering and washing with water.

E.T.

Alkylaminoanthraquinone Dyes for Acrylonitrile Fibres
FBy *BP* 807,241

Alkylaminoanthraquinones which contain in the alkyl radical either tertiary amino or quaternised ammonium groups, e.g.—



dye and print polyacrylonitrile fibres from neutral, acid or slightly alkaline baths with excellent fastness to light and very good to washing. They are made by replacing anthraquinone-α-groups, e.g. Hal, NO_2, OH, OAlk, OAryl, with amines of formula—

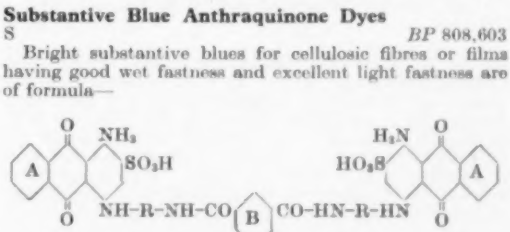
$$R^1HN - X - NR^2R^3$$

(X = divalent aliphatic radical; R^1 = H, Alk; R^2R^3 = Alk or form a ring with the tertiary N). Condensation occurs at 60–120°C. in organic solvents in presence of acid-binding agents and Cu catalysts with such amines as 1-dimethylamino-2-aminoethane, *N*-(β-aminoethyl)-piperidine etc. Quaternisation is effected by treating the amine with alkyl or aralkyl halogenides or sulphates.

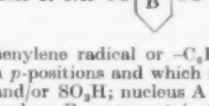
E.T.

Substantive Blue Anthraquinone Dyes
S *BP* 808,603

Bright substantive blues for cellulosic fibres or films having good wet fastness and excellent light fastness are of formula—



(R = *p*-phenylene radical or $-C_6H_4COHNC_6H_4-$ having valencies in *p*-positions and which may be substituted by lower Alk and/or SO_3H; nucleus A may contain SO_3H or 1–2 Hal; nucleus B may contain lower Alk, NO_2, NH_2, acylamino, SO_3H). They are made by condensing aq. solutions of alkali-metal salts of—

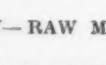


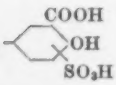
with a benzene-1,3-dicarboxylic acid halide (1 mol.) dissolved in, e.g. CCl_4, $CHCl_3$, toluene, etc. at pH 7–9 (maintained by addition of, e.g. Na acetate, Na_2CO_3 etc.) and at 20–50°C. 17 examples are given.

E.T.

Dyes containing Substituted Sulphonamido Groups
FBy *BP* 807,224

Phthalocyanine, anthraquinone, dioxazine or azo dyes containing ≮ 1 sulphonic acid amide group having ≮ 1 replaceable H atom attached to the amide N are reacted with alkylating agents to give products with better fastness properties than the starting substances. These products contain the group(s)—

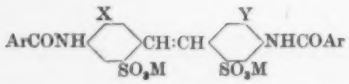


(R¹ = a  group; R² = aliphatic radical).

Thus, Cu phthalocyanine-(3)-sulphotrichloride (1 mol.) is reacted with 5-amino-3-sulpho-2-hydroxybenzoic acid (1·5 mol.) and then with 1-diethylamino-2-chloroethane. E.T.

Alkoxy Diaminostilbenedisulphonic Acids—Fluorescent Brightening Agents
American Cyanamid Co. *USP* 2,848,484

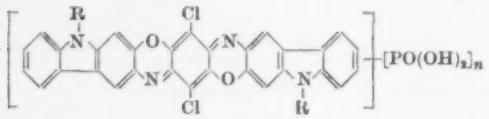
Compounds of formula—



(X = alkoxy; Y = Cl or alkoxy; M = H, alkali metal or NH_4) are fluorescent brightening agents having affinity for cellulose. The compounds particularly specified are disodium 4,4′ - bis- (2,4 - dimethoxybenzamido) - 5,5′- di - methoxystilbene-2,2′-disulphonate, disodium 4,4′-bis-(2,4 - dimethoxybenzamido) - 5 - methoxy - 5′- chlorostilbene-2,2′-disulphonate and disodium 4,4′-bisethoxybenzoylamino) - 5 - methoxy - 5′ - chlorostilbene - 2,2′ - disulphonate. They are applicable from soap solution. In acid baths they also show affinity for nylon, wool, and silk. C.O.C.

Phosphonated Dioxazine Dyes
General Aniline *USP* 2,845,420

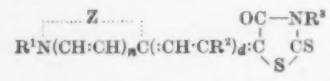
Bright blue water-insoluble dyes of excellent light fastness applicable to nylon from an aq. organic solvent/$Na_2S_2O_4$ vat are



(R = H, lower Alk, haloalk, cyanoalk, hydroxyalk, *cyclo*alk, aralk. aryl; *n* = 1–2). The dioxazine dyes are made, e.g., by reacting tetrachloroquinone (1 mol.) with 3-amino-9-ethylcarbazole (2 mol.) in 95% ethanol containing Na acetate. The product is ring-closed by $C_6H_5SO_2Cl$ treatment, refluxing with nitrobenzene and $K_3Fe(CN)_6$ or C_6H_5COCl and MnO_2. Phosphonation is effected by dissolving in tetraphosphoric acid (phospholeum) containing 83–84% P_2O_5 and heating at 100–200°c. to completion. Dilution with water precipitates the product which is then filtered. The dyes are vatted using $Na_2S_2O_4$ in mixtures of water and 25–85% of organic solvent, e.g. picoline, formamide, pyridine, etc. E.T.

Merocyanine Dyes
Kodak *BP* 807,460

Dyes of formula—



(*n* = 0 or 1; *d* = 0, 1 or 2; Z = atoms to complete a 5- or 6-membered heterocyclic nucleus; R^1 = subst. or unsubst. Alk or allyl; when *d* = 1 then R^2 = H, Alk of 1 or 2 C or alkoxy group of 1 or 2 C; when *d* = 2 then R^2 = H; R^3 = pyridyl or quinolyl), e.g. 5-(3-ethylbenzoxazolin-2-ylidene-ethylidene)-3-(2-pyridyl)rhodanine, have photosensitising properties. C.O.C.

Leuco Sulphuric Esters of Vat Dyes
General Aniline *USP* 2,842,552

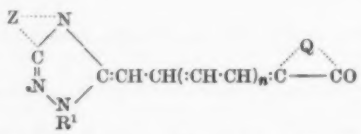
The leuco sulphuric esters are formed by reducing the dyes with, e.g., Cu or Fe in presence of a complex of a tertiary N base, e.g. pyridine, and an esterifying agent, e.g. $ClSO_3H$. The melt is poured into water and alkali; the N base is recovered by vacuum distillation; metal residues are removed by filtration and the filtrate is salted to give the product. Yields are often only 60% theory so that, especially with leuco esters of low solubility, it is necessary to cool the liquor left after vacuum distillation, filter and then extract the product from the mixture of product and metal residues. Extraction by re-slurrying and heating is uneconomical as (*a*) large volumes of liquid are handled, (*b*) an extra filtration is needed, (*c*) complete extraction requires temperatures causing risk of decomposition. Yield increases of 10–40% are claimed, when using a water-soluble N base, if the melt is poured into an amount of water giving a final tertiary N base concentration of 10–30%, an equiv. reduced dye concentration ≯ 5% and if the water contains 1–5% of a hydrocarbon sulphonate of the benzene or naphthalene series or an alkylolamine of 2–3 C or a mixture of these types. These conditions increase solubility so that filtration at 25–40°c. followed by vacuum distillation and salting gives good yields. E.T.

Fluorescent Brightening Agents
FBy *BP* 808,113

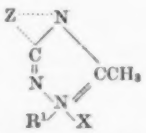
The bluish fluorescent 1,3-diaryl or 1,3,5-triaryl pyrazoline compounds that contain ≮ 1 sulphonamide group attached to the aryl radicals by the S atom, particularly those where the sulphonamide group is in the *para* position of the aryl radical linked to the N atom, are excellent fluorescent brightening agents. Thus 0·1% of 1-*p*-sulphonamidophenyl-3-*p*-chlorophenyl pyrazoline added to a washing powder used on polyamide fibres, acetate rayon or wool having a yellowish appearance turns them pure white. C.O.C.

Merocyanine Dyes from Triazolo Bases
Eastman Kodak Co. *USP* 2,852,384

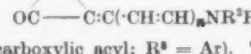
Dyes of formula—



(R^1 = Alk; Z = atoms to complete a benzothiazole or quinoline nucleus; *n* = 0 or 1; Q = atoms to complete a 5- or 6-membered heterocyclic ring) are obtained by condensing a compound of formula—



(Y = acid radical) with one of formula—



(R^3 = H or carboxylic acyl; R^4 = Ar). Thus 6-ethoxy-1-methyl-*s*-triazolo[3,4-*b*]benzothiazole heated with diethyl sulphate and then condensed with 5-acetanilidomethylene-3-ethylrhodanine in presence of pyridine and triethylamine yielded the orange 5-[(6-ethoxy-2-ethyl-1(2*H*) - *s* - triazolo[3,4 - *b*]benzothiazolidene)ethylidene] - 3 - ethylrhodanine. C.O.C.

Low-viscosity Stabilised Vat Dye Pastes
General Aniline *USP* 2,852,331

Vat dyes, and in particular C.I. Vat Blue 6, are obtained as stable aqueous pastes of low viscosity by use of an anionic dispersing agent if 0·5–5·0% on the weight of the paste of dibutyl phthalate, amyl acetate, octyl phenol, caprylic acid, epichlorohydrin or oleic acid is present. C.O.C.

Obtaining Carotene (C.I. Natural Yellow 26) from Vegetable Material
H. M. Barnett, M. L. Hartmann, R. C. Mosher, and H. M. Espoy *USP* 2,848,508

Carrots or other carotene-containing vegetables are pulped and the pulp filtered to remove fibrous material. The filtered juice is then coagulated by heating it in presence of an inert extender, e.g. diatomaceous earth, and the aqueous phase which separates removed. Without further drying the coagulated juice is treated with a volatile water-immiscible solvent, filtered, the water removed and the solvent evaporated to yield a solution of carotene in carrot oil. This solution on cooling yields carotene crystals and a concentrated solution of carotene in carrot oil. C.O.C.

Red Food Dye
Dr. August Oetker *BP* 808,122

Vegetable substances containing a natural red dye, e.g. red beetroot, red cabbage, elderberries, and bilberries, are ground in a colloid mill at room temperature and in absence of added water, into paste. The juice expressed from this paste is immediately filtered and concentrated either as a liquid or a powder. Any odorous or taste-giving substances present can be completely eliminated by steam distillation and spray drying of the extract. It is preferable that the juice be brought to pH 5 by addition of citric or tartaric acid before it is filtered. It is also preferable that gelatin be added to the filtered juice to protect the anthocyanin dyes against external influences such as moisture. C.O.C.

Carbon Black (C.I. Pigment Black 7)
Phillips Petroleum Co. *USP* 2,852,345

In making Furnace Black reduction in the deposit of carbonaceous material around the inlet of the hydrocarbon to the furnace and in the destruction and temperature of the furnace parts can be effected together with increase in the amount and quality of pigment produced, by injecting gaseous hydrogen adjacent to the inlet for the hydrocarbon and/or free-oxygen containing gas inlets.

USP 2,852,346

Plant which provides maximum radiant surface area with the reactor without obstructing the flow of gas through it. C.O.C.

Furnace Black (C.I. Pigment Black 7)
Columbian Carbon Co. *USP* 2,851,337

Method of uniformly mixing the hydrocarbon make with the hot blast flame gases with great rapidity. The more rapid and thorough the mixing the smaller are the particles of the resulting pigment. C.O.C.

Separation of Dry Carbon Black (C.I. Pigment Black 7) from Effluent Furnace Gas
Columbian Carbon Co. *USP* 2,851,124

Wet Oil-Water Emulsion Pelleting of Carbon Black (C.I. Pigment Black 6 and 7)
Phillips Petroleum Co. *USP* 2,848,347

Surface-modified Silica—Flatting Agents for Lacquers and Reinforcing Fillers for Rubber
Monsanto *BP* 807,452–4

When a hydrophilic, amorphous water-insoluble hydrated silica or silicic acid containing a large number (e.g. > 1000) of $>$Si·OH groups and having an acid number of 0·85–5·0 (i.e. mg. of KOH required to bring 1 g. of the material to pH 5·2) is treated with an organic silicate having at least two alkoxy, aryloxy, aralkoxy or alkaryloxy groups or at least one end of two such groups attached to Si which is attached to at least one other O atom, the product obtained is hydrophilic, partly hydrophilic but non-organophilic, partly hydrophobic and organophilic or completely hydrophobic and organophilic depending primarily upon the amount and nature of the organic silicate used. Thus silica aerogel ground in presence of tetraethyl orthosilicate has the same physical properties and acid number as the original aerogel and is partly hydrophobic but non-organophilic. Used as a flatting agent in cellulose nitrate lacquer it does not aggregate on standing but gives a soft settling voluminous precipitate which is readily redispersed by simple stirring. Some of the products are useful as reinforcing fillers for rubber. C.O.C.

Pelleting of Carbon Black (C.I. Pigment Black 6 and 7)
Godfrey L. Cabot *USP* 2,850,403

Vastly improved pellets are produced by any wet pelleting process if the pigment is wetted with water containing $\geq$ 0·4% by weight on the pigment of a soluble hydrocarbon, the wet pellets formed being dried at a temperature and for a time sufficient to completely carbonise the hydrocarbon. C.O.C.

Red Cadmium Pigments containing Mercury
Harshaw Chemical Co. *USP* 2,850,400

A red pigment comparable in quality and tone to cadmium sulphoselenides (C.I. 77202) is obtained by calcining mercury sulphide and cadmium sulphide with an alkali metal sulphide.

USP 2,850,401

Cadmium sulphide and an alkali thiosulphate or dithionite when calcined with a mercury compound which reacts with the thiosulphate or dithionate at the temperature of calcination to yield mercury sulphide, e.g. mercuric chloride, yield a red pigment.

USP 2,850,402

Mercury sulphide, cadmium sulphide and an alkali metal sulphide are made into an aqueous slurry at pH 7·5–12·0 and then filtered until a filter cake is obtained in which the molecular ratio of retained alkali metal sulphide to cadmium sulphide is 0·001–0·05. The cake is then dried and calcined, preferably in an atmosphere of S, and finally washed to remove soluble salts. The hue of the product varies from bright yellowish red when the ratio of mercury sulphide to cadmium sulphide is 0·02 to dark reds when the ratio is 0·25. C.O.C.

Purification of Precipitated Silica
Columbia-Southern Chemical Corpn. *BP* 808,129

Filterable silicas containing 0·5–5·0% by weight of alkali metal ion (reckoned as M_2O) may be purified by mixing them, in presence of water, with a powdered ion exchange resin of particle size larger than that of the silica. The resin is then separated from the resulting slurry and the treated silica scoured free from resin. Either cations or anions can be removed by use of the appropriate resin. To ensure complete removal of metallic cations it is advisable to use enough cation exchanger or to supplement it with another acid to bring the slurry to pH < 5. C.O.C.

Beading or Pelleting of Synthetic Siliceous Pigments
C. A. Greene and E. E. Greene *USP* 2,848,311

The filter cake pulp is tumbled while the moisture content is gradually reduced to about 5% by weight by means of moderate heat and passage of warm dry air across the pulp. C.O.C.

Purifying Silica (C.I. Pigment White 27)
Columbia-Southern Chemical Corpn. *USP* 2,854,316

Ions are effectively removed from precipitated silica by treating it with an ion exchange material or resin whose particle size is greater than that of the flocs or agglomerates of the silica. The mixed slurry is then separated by passing it through a sieve or sifting zone whose perforations or pore size are such that silica passes through and the resin is retained by the screen. The screen is continuously shaken to ensure that the resin particles move along it laterally to its edge and so clogging of the screen is prevented. C.O.C.

Preventing Pigments Caking during Filtration and Improving the Ease with which they may be Ground or Dispersed
Armour and Co. *USP* 2,852,406

If an aqueous precipitate of a pigment is treated so that the particles are coated with a water-insoluble diacid salt of a *N*-aliphatic alkylenediamine then the particles will not agglomerate on filtering and the time needed to grind or incorporate them into paints is much reduced. Thus to aqueous iron blue (C.I. Pigment Blue 27) is added *N*-tallow-trimethylenediamine diacetate and then the stoichiometric amount of Na *N*-coco-aminobutyrate to produce on the pigment particle a coating of 0·5–4·0% by weight of *N*-tallow-trimethylenediamine disodium-*N*-coco-aminobutyrate. The pigment is then filtered off and dried at 90–110°c. C.O.C.

Titanium Dioxide (C.I. Pigment White 6)
Fabriques de Produits Chimiques de Thann et de Mulhouse *BP* 809,284

Titanium dioxide pigments are formed into aggregates of a size which facilitates mixing with other pigments and dispersion in paint vehicles or enamel frits and, in addition, prevents dusting, by dispersing the titanium dioxide in a liquid in absence of an agglutinating agent and then spray-drying in hot gas. C.O.C.

Zeolitic Pigments
J. M. Huber Corpn. *USP* 2,848,346

White pigments of extremely fine particle size (< 0·5 μ.) are obtained by mixing a dilute aqueous solution of an alkali metal silicate with an aqueous slurry of precipitated silica suspended in a dilute aqueous solution of an Al salt of a mineral acid. The mixture is kept alkaline and vigorously agitated. They are used in rubber, paper coatings, inks, paints, and plastics. C.O.C.

Pigment Metal Pastes
English Metal Powder Co. *BP* 808,313

Pastes of high pigment metal content are obtained by (1) blade-stirring, preferably for several hours, a metal paste of non-volatile content 63–68% by weight, if desired with addition of a leafing agent, and (2) mixing the treated paste with further metal flakes, and if necessary some volatile liquid hydrocarbon, so that the final paste has a volatile content of 23–30%. Other pigments or dyes may be added during the second stage. C.O.C.

Reactive Dyes: Synthesis of Dyeings within the Fibre, Solubilities, and Stabilities (VIII p. 231)
Relationship between Structure and Technical Characteristics of Anionic Dyes (XII p. 235)

V—PAINTS; ENAMELS; INKS

PATENTS

Temporary Marking for Laundry Purposes
National Marking Machine Co. *USP* 2,848,348

A marking liquid consists of a fluorescent pigment dispersed in a solvent solution of a colourless resin, e.g. New Jersey Zinc's fluorescent pigment No. 2100, methyl methacrylate resin, toluene, diethylene glycol monobutyl ether acetate and, as dispersing agent, Al stearate. Such an ink leaves colourless impressions which, even in presence of fluorescent brightening agents, show up vividly under ultraviolet radiation but which are effectively and permanently quenched or extinguished by 10–20% formic or acetic acid. C.O.C.

Printing Fluid containing a Red Dye and Crystal Violet Lactone
National Cash Register Co. *USP* 2,850,395

An ink contains a colorant which makes a mark by its own intrinsic colour and a colour-reacting material which, without perceptible delay, assumes a distinctive colour only when applied to a sensitised surface. Thus on sensitised material the ink produces a colour resulting from a mixture of the colour of the two component colorants and which is distinctly different from that produced on unsensitised material. Thus an ink containing Sudan III (C.I. Solvent Red 23) and Crystal Violet lactone when applied to material coated with attapulgite produces a dark purplish black mark compared with the red mark produced on material inert to Crystal Violet lactone. C.O.C.

Non-solid Erasable Writing Material
G. A. Goessling *USP* 2,852,397

A colloidal dispersion of pigment in a lubricated or oily carrier of predetermined viscosity, adhesive and cohesive characteristics is used in a ball point pen to yield impressions which are as readily erasable as those produced by conventional lead pencils. C.O.C.

Incorporation of Pigments into Dextran
Commonwealth Engineering Co. of Ohio *BP* 807,234

Dextran, pigment and water are treated together on hot mixing rolls one of which is at > 100°C. and the other at < 100°C., the mass when thoroughly mixed being stripped from the cooler roll. To prevent the mixture sticking to the hotter roll water is dripped onto the surface of the roll close to the point at which the mixture comes in contact with it. C.O.C.

Ink for Ball-point Pens
G. A. Goessling *USP* 2,852,398

A pigment and/or dye is dispersed in an asphaltum or tarry mass to which has been added a light oil to control erasability and penetrability of the ink. C.O.C.

Phthalocyanine Pigment Cellulose Nitrate Lacquers
Pittsburgh Plate Glass Co. *USP* 2,851,371

Addition of benzyl cellulose prevents flocculation or separation of the phthalocyanine pigment and striation of the lacquer. C.O.C.

Wrinkle Drying Coating Composition
Midland Chemical Corpn. *USP* 2,853,458

A composition which when applied and dried produces a wrinkle finish having very fine and uniform wrinkles consists of an oleoresinous varnish made up of 15–35% fish oil and 85–65% conjugated double-bond vegetable drying oil dissolved in a suitable solvent. C.O.C.

Fluorescent Coating Compositions
Switzer Brothers *USP* 2,851,424

Fluorescent dyes and brightening agents are usually of poor light fastness in solvents, resins or gums. If, however, they are dissolved in solutions or dispersions of amide-aldehyde precondensates which are then cured, the resulting fluorescent composition has a fluorescent life of 10–100 times that expected in the past. C.O.C.

Multilayer Pigmented Finishing Systems for Metal
DuP *USP* 2,847,323

The conventional metal primers used under alkyd resin enamel are not suitable for use under acrylate lacquer topcoats. A suitable primer for such topcoats consists of a baked layer of a liquid coating composition made up of pigment, solvent and, as the principal film-forming material, epoxyhydroxypolyether resin esterified with 10–50% of its weight of drying-type fatty oil acid.

BP 807,912

Conventional primers can be used under acrylate lacquer topcoats if an intermediate undercoat consisting of lacquer grade cellulose nitrate, containing 0–50% of its weight of plasticiser and 0–45% of pigment in total weight of cellulose nitrate and plasticiser, is interposed between the primer and the acrylate lacquer.

BP 807,914

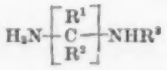
A phosphated copolymer of methyl methacrylate and glycidyl methacrylate can be similarly used as an intermediate undercoat.

BP 807,913

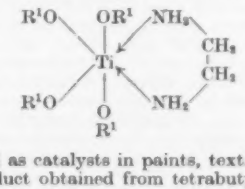
A repair primer for use under acrylate lacquers consists of (*a*) 5–70% cellulose nitrate, 5–75% shellac, 3–40% polyvinyl butyral resin and 0–50% plaster, (*b*) 10–180% by weight of pigment on the total film-forming material, and (*c*) solvent. C.O.C.

Titanium Ester Complexes
National Lead Co. *USP* 2,850,512

The products obtained by treating a monomeric organotitanium ester with a 1,2-diamine, particularly one of formula—



(R^1 and R^3 = H or Alk of 1 or 2 C; R^3 = H, Alk of 1 or 2 C, or amine- or hydroxyl-substituted Alk of < 7 C), e.g. ethylene diamine, have ring structures of the type—



They are used as catalysts in paints, textile finishes, etc. Thus the product obtained from tetrabutyl titanate and ethylene diamine when used as catalyst in a solution of equal parts of Epon 1007 (an epoxy resin), diacetone alcohol and ethylene glycol monoethyl ether when applied to a steel panel and air dried at room temperature for several days yielded a clean, transparent, hard yet flexible film whereas in presence of tetrabutyl titanate and absence of ethylene diamine the resin solution sets immediately to a rubbery semi-solid. C.O.C.

Pearlescent Coating Composition
Sherwin-Williams Co. *USP* 2,851,370

A multiple reflective coating composition comprises a film-forming binder and a lustring agent or pearlescent pigment in lamellar plates, the ratio of pigment to binder being by weight 3:1 to 7:1. Various inorganic materials having a lamellar plate structure, the diameter across the face of the plates being 1–100 μ., e.g. mica, lead acid phosphate, lead acid arsenate, zinc phosphate, lead pyrophosphate, zinc ammonium phosphate, and magnesium ammonium phosphate, are suitable as the pearlescent pigment. C.O.C.

Pigment Metal Formulations
Aluminum Co. of America *USP* 2,848,344

Addition of a small quantity of nitroethane, 1-nitropropane, 1- or 2-nitrobutane, nitrobenzene, *o*-nitrotoluene or 3-nitro-1,2-dimethylbenzene prevents trace-quantities of included water from causing pressure to develop in sealed tins, etc. of inks, paints, etc. containing pigment metals. C.O.C.

Coating with Aqueous Emulsions of Bitumen
Gunac *BP* 809,272

An aqueous emulsion of bitumen and poly(vinyl acetate) yields a strongly adhesive and flexible coating which when dry can be topcoated with a pigmented aqueous emulsion of poly(vinyl acetate). The bitumen does not tend to bleed into the top coating. C.O.C.

Drier Accelerators (III p. 222)
Mica (C.I. Pigment White 20) in Paints (IV p. 222)
Surface-modified Silica— Flatting Agents for Lacquers and Reinforcing Fillers for Rubber (IV p. 227)
Wrinkle Coating or Film-forming Composition (XIII p. 236)

VI— FIBRES; YARNS; FABRICS

Wear Resistance of Cotton and Linen Fabrics. I— Theory of Wear
A. Parisot *Bull. Inst. Text. France*, (75), 7–26 (June 1958)

Each fibre in the testing fabric has an initial mean deformation, determined by the construction of the fabric, which reduces the total work of rupture, represented by the area under the load–elongation (*L–E*) curve. This concept can be carried over qualitatively to the fabric as a whole. Pure mechanical wear involves loss in strength without change in the course of the *L–E* curve; pure chemical wear involves a change in the course of this curve. These ideas are applied to explain the dependence of the wear of cotton and linen fabrics on structure and chemical treatment. J.C.F.

Change of Cross-sectional Area of Single Jute Filaments with Relative Humidity
A. C. Chakravarty
Text. Research J., **28**, 878–880 (Oct. 1958)

Changes in cross-sectional area, determined by cross-width measurements, as a result of sorption of moisture closely resemble the moisture regain isothermals. Hysteresis is prominent only at higher values of R.H., and the small difference between the two curves can be explained by the hypothesis of "transient capillaries". Removal of hemicelluloses depresses the overall swelling of jute, but removal of lignin has no apparent effect. S.B.D.

Cotton–Rayon Blends
W. Bandel and E. Simon
Melliand Textilber., **39**, 1386–1389 (Dec. 1958)

Load-extension diagrams show that 33% of rayon admixed to cotton has little or no detrimental effect on quality of shirtings and may have not only economic but also some processing advantages. S.M.J.

Fat Content of Textiles
M. Kehren and E. Wichter
Z. ges. Textilind., **60**, (19), 860–861 (Oct. 1958)

Data on the extraction of wool yarn and roving with different organic solvents are given. With few exceptions, the amount of material extracted increases with the solvent used in the following order— light petroleum, 1:1 light petroleum–diethyl ether, diethyl ether, trichloroethylene, carbon tetrachloride, methylene dichloride, 1:1 benzene–ethanol, methanol. J.C.F.

Saturation Regain of Viscose Rayon
M. L. Staples *Text. Research J.*, **28**, 950–955 (Nov. 1958)

The moisture regain of viscose rayon in a saturated atmosphere has been estimated from measurements of the longitudinal swelling of the fibre at R.H. ranging from dryness to water immersion. The density of viscose rayon as a function of regain has also been determined. From these data, the volume swelling and the swelling of the cross-sectional area have been calculated. The latter values, at the saturation regain values deduced from the axial swelling measurements, are in good agreement with values found by quite different methods. S.B.D.

Penetration of Electrolytes into Cellulose Fibres— I
B. Andersson and O. Samuelson
Svensk Papperstidning, **61**, 1001–1009 (15 Dec. 1958)

Experiments were carried out with cord, supercord, and rayon at various concentrations and centrifugal speeds. Supercord swelled less than the other rayon samples. The fact that the values obtained by the centrifuge method are dependent upon centrifuge conditions (e.g. r.p.m.) may be explained by variations in the amount of soln. which sticks to the surface of the fibre. Apparent water absorption was determined and real fibre swelling calculated using radioactive polymetaphosphate. Electrolytes investigated were— hydrochloric acid and the chlorides and thiosulphates of Na, K, and Li. R.A.

Stability of Lanthionine Residues in Alkali-treated Wool
M. Van Overbèke, G. Decroix, and G. Mazingue
Bull. Inst. Text. France, (76), 37–48 (Aug. 1958)

The cystine of wool may be divided into two fractions which possess considerably different reactivities towards alkali. It is probable that both fractions react by the same mechanism to produce lanthionine. The severity of the alkaline treatment required for reaction of the less reactive cystine fraction produces progressive destruction of the lanthionine already present, probably with the formation of aminoacrylic acid residues. J.C.F.

Introduction of Amide Cross-linkages into Wool
J. A. Maclaren
Text. Research J., **28**, 946–948 (Nov. 1958)

Peptide-forming reagents have been applied to wool in an attempt to convert "salt-links" into amide cross-links. Wool which has been modified with bicyclohexylcarbodi-imide is almost unchanged in weight, handle, and appearance, but has increased resistance to attack by alkali, alkaline thioglycollate, urea–bisulphite, and trypsin. The mechanism of the changes involved is not known. The extraction of wool with alkaline thioglycollate has been developed as a standard test. S.B.D.

Table of Polyamide Fibres
P.-A. Koch *Teintex*, **23**, 782–811 (Nov. 1958)

Influence of Water Content of Solvents on Viscosity Parameters for Poly-6-caproamide
F. Rybníkář
Faserforsch. und Textiltech., **9**, 500–504 (Nov. 1958)

The influence of the water content of tricresol, sulphuric acid, and formic acid on values of the intrinsic viscosity and Huggins's *k* is studied. An empirical equation allows comparison of results obtained using solvents of differing water content. W.R.M.

PATENTS

Maize Gluten Fibres
Corn Products Refining Co. *USP* 2,854,345

Spray-dried maize gluten is made into an aqueous slurry at < 60°c. The pH is then raised from about 3·5–4·5 to 6·8–7·2 and the slurry vigorously agitated for 1 hr. at 55–60°c. It is then cooled and washed with water by decantation. The washed slurry is then dispersed in aqueous alkali to produce a dispersion of pH 10·5–11·5 which is then filtered and deaerated to yield a translucent solution. This is then extended into a coagulating bath and the filaments formed, stretched, treated with HCHO and baked. The resulting filaments have high resistance to hot aqueous baths. C.O.C.

Colour-stabilised Acrylonitrile Polymers
Chemstrand Corpn. *USP* 2,850,472

A mixture of an organic metal sulphoxylate and benzene phosphorous acid or benzene phosphonic acid stabilises acrylonitrile polymers and their solutions to change in colour on long standing or exposure to heat. C.O.C.

Colour-stabilisation of Acrylonitrile Polymers
Chemstrand Corpn. *USP* 2,849,413
Addition of a mixture of an organic metal sulphoxylate, zinc dicetyl dithiophosphate and an inorganic acid of ionisation constant $> 1 \times 10^{-4}$ to solutions of acrylonitrile polymers enable them to withstand long periods of storage or exposure to heat without change of colour occurring. C.O.C.

Polyacrylonitrile Fibres
FH *USP* 2,846,423
Copolymers of acrylonitrile containing basic groups when treated with hydrofluoboric acid or with a water-soluble salt thereof have much less tendency to discolour when heated, especially at $> 150°$c., lose less in strength and have only slight tendency to become glossy on ironing. The treatment does not otherwise affect their properties, including affinity for dyes. C.O.C.

Diamino-1,2,4-triazoles as Components of Cold-drawn Fibres together with Acrylonitrile and Acrylic or Methacrylic Amides
Koppers Co. *USP* 2,850,485
The dyeing properties of acrylonitrile polymers are much improved if the polymerisation mass contains an amide of acrylic or methacrylic acid and a diamino-1,2,4-triazole. In addition the amides of acrylic or methacrylic acid and diamino-1,2,4-triazoles can be copolymerised by themselves to form useful polymers. C.O.C.

Copolymers of Dialkylaryl Amides of β-Cyanoacrylic Acid or β-Cyanomethacrylic Acid with Acrylonitrile or other Ethylenic Compound
Koppers Co. *USP* 2,850,486
The dyeing properties of acrylonitrile polymers are improved if a dialkylamino aryl amide of β-cyanoacrylic acid or β-cyanomethacrylic acid forms part of the polymerisation mass. These amides also form useful polymers with other compounds containing the $CH_2{:}C\langle$ group. C.O.C.

Copolymers of Acrylonitrile and the Monoamide of Guanazole and β-Cyanoacrylic Acid
Koppers Co. *USP* 2,850,487
The dyeing properties of acrylonitrile polymers are much improved if the polymerisation mixture contains the monoguanazolide of β-cyano acrylic or β-cyano methacrylic acid. C.O.C.

Synthetic Resin Fibres having Antistatic Properties
Union Carbide Corpn. *BP* 808,624
Incorporation of a hydroxyalkyl polyvinyl alcohol containing $< 1{\cdot}5$ parts by weight of alkylene oxide residues per part of polyvinyl alcohol residues in hydrophobic resins confers antistatic properties on fibres formed from them. Thus to a copolymer of 20–50% of acrylonitrile with vinyl chloride is added 5–20% of a hydroxy alkyl polyvinyl alcohol containing 1·5–20 parts of alkylene oxide per part of polyvinyl alcohol. The resulting material when spun from an organic solvent solution and stretched at 110°c. yields fibres having good antistatic properties. C.O.C.

Stabilising Materials containing Polyvinylpyrrolidone to Light and Heat
Dow Chemical Co. *BP* 807,455
Materials, e.g. acrylic fibres, containing polyvinyl pyrrolidone, are stable to light and heat if 0·1–5·0% by weight of zinc formaldehyde sulphoxylate is added to the polyvinyl pyrrolidone before it is incorporated into the fibre composition or the like.

BP 807,456
The same effect is obtained by adding 0·25–5·0% by weight of zinc formaldehyde sulphoxylate to fibre compositions containing polyvinylpyrrolidone. C.O.C.

Yarns for Producing Loop Piles
Collins & Aikman Corpn. *USP* 2,849,785
A number of heat shrinkable ends are X-twisted around a non-shrinkable core. When the resultant composite yarn is heated, e.g. after being used as the pile yarn in a fabric, the outer components of the yarn shrink and so form the non-shrinkable core into irregular pile loops firmly held in place by translucent shrunken ends which being placed low on the loops are hidden from view. C.O.C.

Metal-coated Glass Fibres (I p. 220)
Antistatic Finish Fast to Washing (III p. 220)
Metallising Silicone-treated Fibres (X p. 234)
Polyurethanes for Coating Cloth and for Producing Fibres and Films (XIII p. 236)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Zeta Potential and the Detergency Process
J. C. Harris *Text. Research J.*, **28**, 912–928 (Nov. 1958)
Zeta potential is the difference in potential between the immovable layer attached to the surface of a solid phase (soil or substrate) and the movable part of the diffuse layer in the body of the detergent liquid, and it is of considerable importance in the mechanism of soil removal and redisposition. It is desirable that the zeta potentials of soil particles and substrate be about equal in order to reduce attractive forces between them. The effects of surface-active agents and electrolytes on the zeta potentials of soils and substrates are given and also the relations between zeta potential, suspension values, critical micelle conc., and detergency values. S.B.D.

Wool Piece Scouring
R. Schmidt *Tint. Ind.*, (41), 251–279 (Aug. 1958)
A review covering water, detergents, pH, machines, and faults. S.R.C.

Dissociation Constants of Hypochlorite Solutions
I. E. Flis
Zhur. priklad. Khim., **31**, 1194–1201 (Aug. 1958)
HClO dissociation constants at 10, 25, 35, and 50°c. obtained by thermochemical methods and potentiometric titration are: $(3{\cdot}23 \pm 0{\cdot}05) \times 10^{-8}$, $(5{\cdot}00 \pm 0{\cdot}06) \times 10^{-8}$, $(6{\cdot}61 \pm 0{\cdot}07) \times 10^{-8}$, and $(8{\cdot}91 \pm 0{\cdot}05) \times 10^{-8}$ respectively. It is found by calculation that, in the conventional bleaching sol., Cl_2O content is negligible, and therefore it is assumed that it does not play any noticeable part in bleaching. The results obtained permit the determination of the relationship between the composition of different hypochlorite soln. and their pH values at 10–50°c., and solve the problem dealing with the conditions which lead to chlorination or oxidation in acid hypochlorite soln. T.Z.W.

Activating Sodium Chlorite Bleach Liquors
G. Rösch *Melliand Textilber.*, **39**, 1355–1362 (Dec. 1958)
The author reviews the chemistry of the decomposition of $NaClO_2$ in organic and inorganic acids, and the limitation of ClO_2 evolution by buffering to control pH, by adding H_2O_2, and by activating with hydrolysable organic esters. New work is then described. Ethyl lactate reduces ClO_2 evolution and $NaClO_2$ consumption, but gives insufficient whiteness to cotton in long liquors, though it is preferable to inorganic acids in continuous steam bleaching. $NH_4H_2PO_4$ also reduces ClO_2 evolution and gives a poorer white or requires more time. As it does not evaporate, good whites can eventually be obtained in long liquors, and in 30–60 min. in continuous processes, more economically (3 g. $NH_4H_2PO_4$ per litre instead of 10 ml. $CH_3{\cdot}CH(OH){\cdot}COOC_2H_5$ per litre). pH values of ca. 5·5 are similar, and ClO_2 can be further suppressed with H_2O_2, not usable with lactate. Regeneration of the $NaClO_2$ liquor is possible. Degrees of whiteness are assessed instrumentally. Corrosion is investigated by tabulating weight losses of 4VA steel (improved by adding $NaNO_3$), of 4 different samples of lead (very poor with acid), and of aluminium, both untreated (very poor) and anodised. $NH_4H_2PO_4$ is mildest, also as far as rubber is concerned, and is cheaper than esters. There are seven illustrations, 12 tables, and 18 references. S.M.J.

PATENT

Stabilised Acid-activated Chlorite Solutions
Elektrochemische Werke München *BP* 808,669
Alkali-metal or ammonium formates are very effective stabilising agents for acid-activated chlorite solutions even 0·1 g. Na formate per litre or 0·15 kg. Na formate per 100 kg. material to be bleached results in the bath having little or no smell and prevents corrosion of chrome-nickel-molybdenum steels. C.O.C.

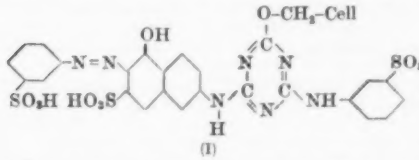
Temporary Marking for Laundry Purposes (V p. 228)

VIII— DYEING

Reactive Dyes: Synthesis of Dyeings within the Fibre, Solubilities, and Stabilities

J. Wegmann

Melliand Textilber., **39**, 1006–1012 (Sept. 1958)



(I)

Complete synthesis of the structure (I) formed by reaction of monochlorotriazinyl (II) dye with cellulose was achieved in aqueous medium. Though cyanuric chloride condensed with metanilic acid reacts with cellulose, the product does not combine with the azo cpd., except in cold dil. acid in < 60 hr. (azo cpd. with free NH_2 groups react faster). Cyanuric chloride reacts with cellulose to form, not the desired dichlorotriazinyl ester, but the hydroxychlorotriazinyl ester (VI) and II-diester. Selective hydrolysis of IV yields hydroxydichlorotriazine (VII), which unlike other dichlorotriazines reacts only sluggishly with cellulose giving VI, owing to lactam–lactim tautomerism. Boiling acidified azo cpd. with VI form I with OH replacing the V residue (VIII). The corresponding hydroxychlorotriazinyl dye (IX) is the end-product of aqueous alkaline hydrolysis of the dichloro cpd. (X). Both Cl atoms of X react with cellulose in cold Na_2CO_3 soln., forming a cross-linked dyeing (XI) insoluble in cuprammonium hydroxide. Dyeings of X developed by steaming are more or less soluble in cuprammonium, one of the ester links of the initially formed XI being hydrolysed to give VIII. The cold-dyeing XI on heating in dil. Na_2CO_3 is rendered soluble in cuprammonium without any bleeding. IX, like VII, barely reacts with cellulose, unless enolisation of the tautomer is enhanced (6% NaOH for 50 hr.), when VIII is formed. The absence of this tautomerism in the amino and methoxyl derivatives accounts for the stability of such dyes in cold Na_2CO_3 soln. and for the loss of reactivity of dichlorotriazinyl derivatives such as X despite retention of one Cl. The greater reactivity of cellulose towards e.g. X compared with that of water is regarded as due to the much lower degree of association of the OH groups in cellulose. A yellow chlorotriazinyl dyeing synthesised on the fibre condensed with a blue aminoanthraquinone dye gives a green dyeing, linking two dyes to cellulose. Reaction with the CH_2OH groups of cellulose as depicted in I is supported by lack of reactivity of alginate and by pptn. with H_2SO_4 or NaOH of fast-dyed cellulose after reacting dyes with soln. of cellulose in cuprammonium, in which the secondary OH groups 2 and 3 are blocked by complexing. Dyeings with reactive dyes forming ether linkages or of the type of II are always soluble in cuprammonium, whereas those of the type of X can react in three ways, giving insoluble di-esters, or soluble monochloro or hydroxy derivatives, only the former of which can be insolubilised. Insolubilisation by cross-linking is also obtainable with one dye containing two II groups. The stability of the dye–fibre linkage depends far more on the carrier grouping than on the dye component; the nature of the former can be diagnosed by the relative ease of acid and alkaline hydrolysis. Dichloro- and hydroxychloro-triazinyl derivatives give dyeings stable towards alkali but not acid; ether linkages such as are formed by chloropropionamides exhibit the converse behaviour, whereas amino- and alkoxy-triazines are intermediate, being relatively stable to both, more so to alkali. These findings are illustrated and the implications are discussed. S.M.J.

Fibre Damage in the Stock-dyeing of Wool

B. Gullbrandson

Text. Research J., **28**, 965–968 (Nov. 1958)

The breaking of fibres on the card resulting from the loss of tenacity of the wool in stock-dyeing processes has usually been ascribed to a damaging effect of the dye. The present investigation shows, however, that most of the reduction in tenacity is due to the setting of curvatures in the packed fibres by the hot-water treatment. S.B.D.

Inhibiting the Fading of Dyes— II

Z. Hajós and J. Fodor

Acta Chem. Acad. Sci. Hung., **16**, 291–9 (1958): *Chem. Abs.*, **53**, 722 (10 Jan. 1959)

When compounds of the type of 3-hydroxy-2-naphthanilide are used as inhibitors for the fading of Benzopurpurin B (C.I. Direct Red 21) the fluorescences and light absorption are proportional to inhibiting effects. One of the essential factors in the inhibiting action of these compounds is their sensitivity to air and CO_2. Derivatives sensitive to CO_2, in which enolisation of the carbo-imide group is hindered, are less efficient inhibitors. Among structurally similar compounds those with higher dipole moments have increased inhibiting action. C.O.C.

Diffusion of Dyes in Cellulose

K. J. Heritage

Trans. Faraday Soc., **54**, 1902–1910 (Dec. 1958)

The integral diffusion for dyes with a low affinity for cellulose is independent of dye concentration except in the most dilute solutions. The permeability of a Cellophane membrane to a dye of low affinity is reduced when the carboxyl content of the cellulose is increased, and with increasing dye affinity the diffusion coefficient decreases correspondingly. Dyes of higher affinity show a linear increase of diffusion coefficient with concentration. An acid dye with a pronounced amphipathic constitution is strongly absorbed by cellulose and differs from other dyes in that the diffusion coefficient falls as the concentration rises. W.R.M.

Vegetable Fibres— High-temperature Dyeing with Direct Dyes

E. Herrmann

Z. ges. Textilind., **60**, (19), 862–865 (Oct. 1958)

In this summarising article, the last of a series, direct dyes are classified according to their stability and exhaustion properties in dyeing vegetable fibres at temperatures above 100°C. The stability of the fibres themselves at high temperatures is also considered. J.C.F.

Oxycelluloses— Carboxyl Content and Behaviour towards Direct Dyes

P. Rochas and C. Daneyrolle

Bull. Inst. Text. France, (76), 21–36 (Aug. 1958)

A study of the dyeing behaviour of acid oxycelluloses derived from viscose rayon towards disazo dyes, and of the interaction in solution between the same dyes and soluble non-ionic or anionic polymers, indicates that viscose rayon behaves as an anionic polymer. Substantivity is greatest for dyes which are least anionic in character. Oxidation of the cellulose reduces by the same amount the degree of fixation of dyes which possess the same number of SO_3H groups. The relative reduction in fixation for the oxycellulose is less the greater is the substantivity of the dye. Dyes containing NH_2 groups have better substantivity than those containing OH, Cl, or O·CH_3. J.C.F.

Chroming of Wool

G. Gianola, O. Meyer, and A. Lantz

Bull. Inst. Text. France, (76), 59–70 (Aug. 1958)

The free amino acids cystine, cysteine, tyrosine, tryptophan, methionine, serine, and histidine reduce $K_2Cr_2O_7$ in acid solution: cystine is oxidised to cysteic acid, methionine to homocysteic acid, and serine to aminomalonic acid. Other common amino acids do not react. Chroming of wool reduces the content of tyrosine (10%), tryptophan (46%), and methionine (38%), but the cystine content and the total sulphur remain unchanged. Chromed wool has a reduced solubility in urea–bisulphite, indicating the formation of interchain cross-links. Dinitrophenylation of chromed wool reduces the rate of extraction of tervalent chromium by H_2SO_4 and oxalic acid, and this is taken as evidence that the NH_2 groups of wool are involved in the cross-links formed on chroming. J.C.F.

Dyeing Cellulose Acetate

A. Schaeffer *Melliand Textilber*, **40**, 75–76 (Jan. 1959)

Concise review of known work on the mechanism of the dyeing of cellulose acetate with disperse dyes (solid solution and some adsorption), basic dyes (adsorption), and acid dyes (solid solution, especially if sulpho groups are few, chelated, or replaced by carboxyl groups). 12 references. S.M.J.

Thermodynamics of Adsorption of Small Molecules by Proteins
S. P. Harrold and B. A. Pethica
Trans. Faraday Soc., **54**, 1876–1884 (Dec. 1958)
An analysis of the binding of small molecules to proteins in solution is given using methods of adsorption thermodynamics. Heats of binding may be obtained as a function of the binding ratio directly for non-ionic molecules and ionic molecules binding in the presence of excess of electrolyte. Data are given for the binding of sodium dodecyl sulphate to keratin, and the theory is applied to calculate heats of binding for concentrations up to the critical micelle concentration. Physical adsorption appears to be responsible for binding. W.R.M.

Effect of Lubricants on Trevira Polyester-Wool Blends
W. Linke *Melliand Textilber.*, **40**, 73–74 (Jan. 1959)
The effect of lubricants and emulsifiers used in worsted spinning is investigated by spotting them on samples of polyester staple fabric, both heat-set and not, and of polyester filament fabric, ageing and heat-treating in various ways, washing (6 recipes), and dyeing with 1% Samaron Blue 3GL (FH). None of the 5 emulsifying agents affect dyeability. Lubricants derived from olive or ground-nut oil produce localised deposits, resistant to scouring, with increased dye uptake, as found by Hartley and Elsworth (*J. Textile Inst.*, **49**, P 554 (1958)). Mineral oils require very thorough alkaline scouring with non-ionic detergents such as Hostapal CV (FH). Low oil contents can still cause trouble by aggregating during dyeing. Olein behaves well, but is rarely used. Faultless results are obtained with the fat-free lubricants Lanigan KG and KGN. S.M.J.

PATENTS

Dyeing Nitrogenous Fibres with Metallised Azo and Acid Milling Dyes and Stripping Such Dyeings
S *BP* 808,647
Wool, silk, polyamide or other nitrogenous fibres are dyed level and fast to rubbing in an aqueous bath at pH 4·0–5·5 in presence of (*a*) a water-soluble polyglycol ether whose hydrocarbon radical is of $>$ 7 C and may contain N and/or O and/or P atoms and/or substituents, (*b*) an organic acid, (*c*) a buffer salt, and (*d*) $<$ 10% on weight of the fibre of an alkali metal sulphate. Dyeing is done at the boil by adding the dye to a bath containing the other ingredients. The dyeings can be stripped by boiling them in a similar bath containing no dye. C.O.C.

Auxiliary Agents for Use in Dyeing Wool and other Polypeptide Fibres
Gy *BP* 809,209
A mixture of 30–90% by weight of polyalkyleneoxyhydroxyalkyl compounds containing a lipophilic radical and $>$ 9 recurring alkyleneoxy groups when mixed and 70–10% of their acid sulphuric acid esters are of much use in acid dyebaths particularly those containing pre-metallised azo and azomethin dyes having no acid dissociating, salt-forming substituents not forming part of the heavy metal-dye complex. C.O.C.

Dyeing Polyamide Fibres with Neutral-dyeing Metal-complex Dyes
Gy *BP* 809,258
Addition to the dyebath of a polyglycol ether of an amino compound of 16–18 C gives much better levelling of polyamide fibres having irregular affinity for dyes. C.O.C.

Assistants for Dyeing Polyesters and Cellulose Triacetate with Disperse Dyes
FBy *BP* 808,948
Presence in the dyebath of an ester of an alkyl analogue of salicylic acid, e.g. methyl *o*-cresotate, results in deeper dyeings than are obtained by use of salicylic esters. C.O.C.

Dyeing Vinylidene Cyanide Interpolymers with Basic Dyes
B. F. Goodrich Co. *USP* 2,848,296
The affinity of vinylidene cyanide interpolymer fibres for basic dyes is much increased if dyeing is carried out in presence of 2-hydroxy-3-naphthoic acid or *N*-*β*-naphthyl-*β*-alanine. C.O.C.

Vat Dyeing
Nederlandse Organisatie voor Toegepastnatuurwetenschappelijk Onderzoek ten Behoeve van Nijverheid, Handle en Verkeer *BP* 808,219
A vat especially suitable for continuous dyeing consists of water, a vat dye, a reducing agent, e.g. Na hyposulphite, $<$ 1 g. mol. of alkali metal hydroxide per litre and sufficient of an aldehyde that the absolute value of the bath potential at room temperature is lower, but at 100°c. higher, than the leuco-potential of the dye at the dye concentration used. Thus a vat is made up of Cibanone Olive S pdr. fine for dyeing (C.I. Vat Black 25) (120 g.), glycerol (200), NaOH (320), 80% Na hyposulphite (320), 40% formalin (276 g.), and water (to bring to 4 litres). The potential of the vat before the dye is added is −885 mv. at 20°c. and −790 mv. immediately after the dye is added. The leuco-potential of the dye at 60°c. at the concentration used in the bath is −1000 mv. Cotton is padded with this bath to a dye pick-up of 3%, it is steamed for 72 sec., oxidised with Na perborate, soaped and dried. This yields a dyeing somewhat deeper than that obtained by dyeing similar material by the conventional method. C.O.C.

Dyeing Tightly Wound Glass Fibres Containing a Water-swellable Size
Owens-Corning Fiberglas Corpn. *USP* 2,849,333
Glass fibres sized with gelatin or other water-swellable material are readily dyed, with complete and rapid penetration, by acid and direct dyes, if the dyebath consists mainly of alcohol in a substantially non-solvent system having a coupler to maintain the stability of the bath, e.g. 1 g. Pontamine Sky Blue 6BX (C.I. Direct Blue 1) dissolved in 3 mol. ethylene glycol and 97 mol. methyl alcohol. C.O.C.

Antistatic Finish Fast to Washing (III p. 220)
Relationship between Structure and Technical Characteristics of Anionic Dyes (XII p. 235)

IX— PRINTING

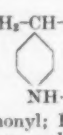
PATENTS

Printing Pastes
Gy *BP* 808,925
Boric acid or other borate ion donor compounds increase the viscosity of starch carrier emulsions and so may be used to control the viscosity of printing pastes to a suitable level for printing. C.O.C.

Diazotype Material
Charles Bruning Co. *BP* 808,527
Paper, cloth or other base is coated with non-cooked and non-swollen rice starch whose particles have been coated with a composition containing a light-sensitive diazo compound, so that they can act as foci for the development of the colour. The coupling component may also form part of the composition. The treated material yields prints of greatly improved smoothness and intensity. C.O.C.

Blue Print Paper
Keuffel & Esser Co. *USP* 2,852,377
Incorporation of silica in the sensitising solution gives at least as good results as does giving the paper a preliminary coating with silica (cf. *USP* 2,433,515) if the amount of silica deposited on the paper from the solution is high enough. C.O.C.

Incorporating Polymeric Colour Formers or Couplers in Photographic Emulsions
Eastman Kodak Co. *USP* 2,852,381
If polymeric colour formers, especially those containing recurring polymeric units free from alkali-solubilising groups and of formula—



(A = carbonyl or sulphonyl; R = a group capable of reacting with the oxidation product of a primary aromatic amine to form a dye), are dissolved in a water-insoluble crystalloidal material of m.p. $<$ 100°c. before being dispersed in the emulsion, they do not separate out either during coating, processing or storage. C.O.C.

Negative Working Diazo Sulphonate Foils
General Aniline *USP* 2,854,338

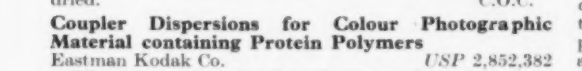
A transparent base is coated with a maleic anhydride–vinyl compound copolymer mixed with a compatible hydrophobic resin. The copolymer is then treated to effect splitting, which may include partial amidification of the maleic anhydride linkages. Finally the resulting water-sensitive layer is treated with a coating solution containing a diazo sulphonate and a coupling component. This yields a cheap, easily processed, water-sensitive layer which is dimensionally stable, permits ready rinsing out of chemicals, is easily handled when wet and is very quickly dried. C.O.C.

Coupler Dispersions for Colour Photographic Material containing Protein Polymers
Eastman Kodak Co. *USP* 2,852,382

When the ratio of coupler solvent to solvent is $< 1:1$, some couplers on development yield dyes whose hue is different from the desired normal hue and which have low contrast and/or low maximum density. This is overcome by including in the emulsion the product obtained by polymerising a protein with a monoethylenically-unsaturated compound containing the –CH:C< group. C.O.C.

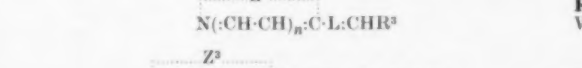
Supersensitising of Emulsions containing Dicarbocyanine Dyes
Eastman Kodak Co. *USP* 2,852,385

Dicarbocyanine dyes, particularly those of formula—



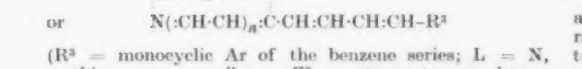
$$R^1N(CH{:}CH)_nC{:}CH{\cdot}CH{:}CH{\cdot}CH{:}CH{\cdot}C{=\!=\!=}NR^2X$$

(R^1 and R^2 = Alk; n = 0 or 1; X = acid radical; Z^1 = atoms to complete a 5- or 6-membered ring; Z^2 = atoms to complete a benzoxazole, naphthoxazole, benzoselenazole, naphthoselenazole, benzothiazole or naphthothiazole nucleus), when mixed with bases of formula—



$$N(:CH{\cdot}CH)_n{:}C{\cdot}L{:}CHR^3$$

or



$$N(:CH{\cdot}CH)_n{:}C{\cdot}CH{:}CH{\cdot}CH{:}CH{-}R^3$$

(R^3 = monocyclic Ar of the benzene series; L = N, methin, cyano or alkoxy; Z^3 = atoms to complete a nucleus) act as photographic supersensitisers, e.g. a mixture of 3,3′-diethyloxathiadicarbocyanine iodide and 2-styrylbenzimidazole. C.O.C.

Colour Photography using Coloured Couplers
Eastman Kodak Co. *USP* 2,852,370

A masking image composed of the uncoupled portion of a coloured coupler containing sulpho or carboxy groups formed by the process described in *USP* 2,808,329 (J.S.D.C., **74**, 720 (1958)) is treated with a basic dye, preferably of different hue from the image. This yields a composite masking image of two dyes which compensates for the unwanted light absorptions of the normal dye image in the emulsion layer. C.O.C.

Multi-colour Screen Film for Use in Additive Colour Photography
Emimeta-Fabrikations-Anstalt *BP* 809,072

Engraving of Printing Rollers (I p. 219)
Colour Couplers (IV p. 222)

X—SIZING AND FINISHING

Effect of Moisture Content and Calendering in Chemical Shrinking of Cotton Fabrics
R. Steidl *S.V.F. Fachorgan*, **13**, 694–702 (Dec. 1958)

Bleached cotton fabrics were treated with Lyofix PR (Ciba), starch, and $(NH_4)_2SO_4$ (4, 2, and 0·3 g./litre respectively). Samples at differing moisture contents (four levels) were finished with and without calendering, and subsequently tested for residual shrinkage (warp and weft directions), tear strength, and resistance to abrasion (before and after repeated washing). The results are presented graphically (32 diagrams). It is apparent that the moisture content and the method of calendering influence residual shrinkage, tear strength, and abrasion resistance. L.A.T.

Development of Textile Finishing since Maria Theresia
G. Schwen *Melliand Textilber.*, **40**, 51–55 (Jan. 1959)

Lecture, delivered on the occasion of the bicentenary of the Vienna Textile Institute, reviewing the development during its existence of fibres, fabrics, dyes, dyeing and printing, auxiliaries, and finishing. S.M.J.

Topochemical Mechanisms in the Preparation and Deacetylation of Partially Acetylated Cottons
A. V. Bailey, E. Honold, and E. L. Skau
Text. Research J., **28**, 861–873 (Oct. 1958)

Data are presented to show that in partially acetylated cottons the amorphous regions are acetylated first and that deacetylations with 2 N-HCl or with 0·5 N-NaOH proceed by different mechanisms. With HCl the amorphous areas are more or less completely deacetylated before the crystalline areas are attacked, but with NaOH both are deacetylated simultaneously, so that deacetylation proceeds annularly into the fibre. The properties of original partly acetylated cottons differ markedly from those of the same acetyl content obtained by deacetylation, those of the latter depending on original acetyl content and the reagent used in the deacetylation. S.B.D.

Iodine Sorption Values of some Chemically Modified Cottons
A. V. Bailey, E. Honold, and E. L. Skau
Text. Research J., **28**, 895–896 (Oct. 1958)

Plotting iodine sorption values against degree of substitution for partially acetylated, cyanoethylated, and β-propiolactoned cottons gives a series of curves with iodine sorption increasing with the degree of substitution. For a given degree of substitution the cyanoethylated samples sorbed more than the acetylated samples, and those prepared with β-propiolactone gave values above or below the others depending on their mode of formation. S.B.D.

Effect of Crease-resist Finish on Crease Recovery—IV
W. Wegener
Melliand Textilber., **39**, 1379–1386 (Dec. 1958)

As previously, various concn. of dimethylolurea resin applied to filament taffeta, in this case of cuprammonium rayon, are examined for dependence of crease recovery on time ($\alpha'_{60} = 0{\cdot}61 + 1{\cdot}323\ \alpha'_5$), direction of crease (max. at 45°), and resin concn. (2% and 7% are similar, 10% better). Present and previous results are compared. Crease recovery decreases with increasing contraction in weaving. 13 graphs, 3 tables. S.M.J.

Wet-crease-resist Finishing of Viscose Rayon
H. A. Wannow
Melliand Textilber., **39**, 1376–1379 (Dec. 1958)

Practical procedure is described in detail, with specific examples and several drip-dry recipes. Dimensional changes in drying, calendering, condensing, and washing off are tabulated for different fabrics, as are also wet and dry crease-recovery angles before and after washing, tensile strengths, and residual shrinkages. Pretreatment in 6° Bé. NaOH at 20°C. improves wet decreasing and wet crease resistance without excessive harshening of handle or reduction of tensile strength. Melamine resins alone and combined with modified urea resins on the one hand, and dimethylolethyleneurea resins when combined with etherified starches on the other hand, are suitable. The latter give much lower residual shrinkages and are faster to washing, but reduce the light fastness of some direct dyeings, and lower tensile strength more. Steel calendering, usual with cotton, lowers crease resistance. The wet crease-resist effect is more marked than with cotton, and the loss of dry tensile strength lower. Dry crease-resistance, though improved, is not very good. Wet tensile strength is raised and fibre swelling lowered, so that drying is accelerated. Wash fastness is very high. S.M.J.

Durable Waterproofing of Wool with Fatty Acid–Chromium Complexes
A. Engeler and W. Schefer
Textil-Rund., **13**, 643–649 (Nov. 1958)

Wetting agents and dry-cleaning water-carriers must be rigorously excluded. Baking must be at $\geq$ 115°C. and for $\geq$ 15 min. to avoid acid damage to the fibre, although organic bases can be added to the impregnating liquor to counteract this. S.R.C.

Serviceability of a Minimum-care Garment

M. A. Morris and B. Wilsey

I— Subjective Evaluation

Text. Research J., **28**, 881–887 (Oct. 1958)

Trousers made from 65% polyester fibre and 35% rayon blended fabric were worn a total of 750 hr. After every 150 hr. of wear, half of these garments were dry-cleaned and half home-laundered. The garments were evaluated by both the wearers and experts after each wear period and after each cleaning. The dry-cleaned and pressed trousers were consistently rated above the laundered and pressed ones. The laundered trousers required pressing before they were acceptable for wear.

II— Laboratory Evaluation *Ibid.*, 887–891

Physical measurements, e.g. of strength, elongation, crease recovery, were made on a minimum-care polyester fibre and rayon blended fabric to determine the effect of maintenance and wear on serviceability. Physical characteristics of the fabric, with the exception of stiffness and abrasion, showed only slight changes. S.B.D.

Titanates in Showerproofing

R. Sidlow *Dyer*, **120**, 301–304 (29 Aug. 1958)

Efficiency of titanates (in conjunction with wax) as showerproofing agents for textiles depends on their hydrolytic degradation. Stability of various titanates and titanium soaps was estimated by exposing 1% solutions in white spirit to an indoor atmosphere. Butyl and the lower alkyl titanates show some precipitation, due to hydrolysis, within a few hours, 2-ethylhexyl and methylcyclohexyl titanates within 1–3 days, and 3,5,5-trimethylhexyl and "iso-octyl" titanates in 2 weeks. Mixtures of the titanates of butanol and hexylene glycol (2-methylpentene-2,4-diol) resemble the iso-octyl ester, while titanium stearate is apparently unaffected after 6 weeks. Data (spray rating, hydrostatic resistance, and Bundesmann tests) on gaberdines treated with paraffin wax and titanates in white spirit show that those titanates which give precipitation in about 2 weeks by the above test provide the most effective proof. Treatment at the rate of 1·6% wt. wax and 0·6% wt. titanate on the fabric is suitable for reproofing dry-cleaned garments. Residual detergent in the fabric reduces the effectiveness of the proof. E.C.

Fate of Thioglycollic Acid in Wool durably creased with Thioglycollate

P. H. Springell *Text. Research J.*, **28**, 874–878 (Oct. 1958)

The effects of various treatments are followed by adding an amount of ^{35}S-labelled ammonium thioglycollate to the solutions used to impart creases. Steam ironing is not associated with any loss of radioactivity, but washing with running water results in a rapid initial loss followed by a much slower decrease. Treatment with sulphide, sulphite, or alkaline cyanide removes radioactivity from the wool, but lithium bromide and urea have little effect on the radioactivity. The findings are discussed in the light of current theories of durable pleating. S.B.D.

Mothproofing of Wool

E. Laibach *Melliand Textilber.*, **39**, 1389–1391 (Dec. 1958)

Eulan U33 (FBy) mothproofs wool without toxic risks. S.M.J.

PATENTS

Cross-linking Reduced Keratin with Polyimides or Polyamides

U.S. Secretary of Agriculture *USP* 2,850,351

Compounds containing < 1 acyl-N group containing an activated carbon-to-carbon double bond are used for cross linking reduced keratin. Thus white flannel was treated for 1 hr. at 50°C. in aqueous 0·4 M. 2-mercapto ethanol containing 0·1% of the iso-octyl phenyl ether of polyethylene glycol (Triton X–100) as a wetting agent, using 30 ml. solution per g. of wool. The flannel was then rinsed in running water and then treated for 1 hr. in 0·1 M. sodium borate buffer at pH 8·0 and 35°C. with 2 millimoles per g. wool of 1,3,5-triacyl hexahydro-s-triazine and then thoroughly rinsed. The treated flannel had a cystine content of 4·0% compared with 11·4% for an untreated sample and supercontraction 0 compared with 28% for the untreated flannel (measured on single fibres by Speakman's method— J.S.D.C., **52**, 335 (1936)). 14 classes of compounds are cited as being suitable. C.O.C.

Size for Nylon

Monsanto *USP* 2,848,357

An aqueous solution of a terpolymer of vinyl acetate, maleic anhydride and styrene used for sizing nylon warps gives good protection during wearing and is readily removed by simple scouring. C.O.C.

Imparting Dimensional Stability to Tubular Knitted Fabrics

Samcoe Holding Corpn. *BP* 808,985

The fabric is spread out flat to the desired width, steamed and then immediately impregnated with a resin precondensate and finally dried and baked. C.O.C.

Improving the Properties of Polyester Fibres

FH *BP* 808,558

The texture, antistatic, and water-repellent properties and, in many cases, resistance to dirt, of polyester fibres are greatly improved by treating them before or after dyeing or heat setting, with a high molecular aromatic polyhydroxyl compound having affinity for hide powder, then, preferably while hot, treating with a dispersion or solution of a 1,2-ethyleneimide or 1,2-ethylene urea containing an aliphatic hydrocarbon residue of > 5 C, and finally drying and baking. Thus worsted-spun polyethylene terephthalate yarn is dyed with a black disperse dye and then treated at 60°C. in an aqueous bath containing 1% tannin on the weight of yarn and brought to pH 5 with oxalic acid. After 20 min. 2% of tartar emetic is added and the yarn treated for a further 20 min. at 60°C. It is then rinsed with cold water and treated for 20 min. at 40°C. in a bath containing 1·5 g./litre of *N*-octadecyl-*N'*-(1,2-ethylene)-urea, centrifuged, dried at 60–70°C. and baked for 60 min. at 100°C. The yarn is rendered softer and more voluminous, water-repellent and less inclined to become dirty and has an antistatic finish which is fast to soaping at 60°C. C.O.C.

Modifying the Properties of Polyethylene Terephthalate Textiles

Vereinigte Glanzstoff-Fabriken *BP* 808,967

Modification of *BP* 805,525 (J.S.D.C., **75**, 128 (Feb. 1959)). The process is more readily and more cheaply carried out if the chlorinated hydrocarbon is used as an aqueous emulsion instead of in the pure form. C.O.C.

Metallising Silicone-treated Fibres

Commonwealth Engineering Co. *USP* 2,847,322

The fibres or fabric are given a protective coating of a flexible heat-resistant silicone and then treated with a gaseous metal compound which decomposes on heating to deposit metal on the fibres. Thus fibres are coated with an organo polysilane–polysiloxane resin and then treated with nickel carbonyl at 315°F. whereby metallic nickel is deposited on the fibres. C.O.C.

Fungicides— Rot and Water Repellent Finish

Phillips Petroleum Co. *USP* 2,848,356

The products obtained by reacting a heavy metal salt of an organic carboxylic acid containing an active halogen atom with a heterocyclic nitrogen base polymer retain the fungicidal and fungistatic characteristics of the heavy metal salts. Applied to textiles they impart a rotproof finish which is more resistant to moisture and weathering than that given by the heavy metal salts and in addition they also confer a high degree of water repellency. Thus cotton duck treated with the product obtained by treating a butadiene–methylvinylpyridine copolymer dissolved in an aqueous mixture of acetic acid and isopropyl alcohol with cupric chloroacetate dissolved in aqueous isopropyl alcohol has excellent water repellency and resistance to *Chaetomium globosum*. C.O.C.

Rotproofing of Cellulose

National Cylinder Gas Co. *USP* 2,850,405

Cellulose is impregnated with Cu acrylate and/or Cu furoate, preferably from aqueous solution, and is then baked at 120–180°C. This gives a rotproof finish which is very resistant to weathering. C.O.C.

Softening of Glass Fibres and Improving their Receptivity for Resins

L–O–F Glass Fibers Co. *USP* 2,854,355

The fibres are treated with an aqueous solution of a hydrolysed vinyl silane at pH 4·5–7·5 and dried in a still atmosphere. C.O.C.

Rotproofing
B. O. Hager *BP* 808,277

The material is treated with a compound of any amine-forming metal dissolved in an aqueous solution of ammonia and CO_2, the CO_2 content being $\leqslant$ 80%, and preferably $\leqslant$ 100%, of the total metal content. When the excess ammonia and CO_2 evaporates after the treatment, a non-leachable compound of the metal is formed in the material and there is no formation of soluble electrolytes. C.O.C.

Bonding Fabrics of Synthetic or Mineral Fibres to Poly(vinyl Chloride)
Fothergill & Harvey (Research & Development) and Vinyl Products *BP* 809,290

Much stronger bonding than that given by the process of *BP* 745,060 (*J.S.D.C.*, **72**, 259 (1956)) is produced if the fabric is impregnated with a dispersion of a mixture of plasticised poly(vinyl chloride), an epoxy resin and a hardener for the epoxy resin in organic medium, dried, cured and then coated with poly(vinyl chloride). C.O.C.

Metal-coated Glass Fibres (I p. 220)

β-Amino Lower Alkyl Carbamate–Formaldehyde Adducts for Improving the Recovery from Creasing and Dimensional Stability of Cellulosic Textiles (III p. 221)

β-Amino Lower Alkyl Carbamate–Epichlorohydrin Adducts for Improving the Recovery from Creasing and Dimensional Stability of Cellulosic Textiles (III p. 221)

Stabilising Materials containing Polyvinyl-pyrrolidone to Light and Heat (VI p. 230)

Wrinkle Coating or Film-forming Composition (XIII p. 236)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Distribution of Substituents in Ethylcellulose—Heterogeneous Reaction with Ethyl Chloride
I. Croon and E. Flamm
Svensk Papperstidning, **61**, 963–966 (30 Nov. 1958)

Two ethylcelluloses have been studied. Cotton linters were converted into alkali cellulose by treatment with 16·4–32·2% NaOH, followed by reaction with C_2H_5Cl in an autoclave. The hydrolysates were separated by carbon-column chromatography, and eight of the components were obtained in the crystalline state. The results were very similar to those obtained with CH_3Cl, but reaction with C_2H_5Cl gives more heterogeneous distribution owing to the larger bulk of the C_2H_5Cl molecule. 32·2% NaOH gave a more heterogeneous product than 16·4%. Results show that 2-*O*-ethylglucose is the predominant mono-*O*-ethylglucose in the hydrolysates and also the most easily eluted from the carbon column. R.A.

PATENTS

Sized Paper
National Dairy Products Corpn. *USP* 2,830,916

The amount of starch used can be substantially reduced by adding 1–15% of its weight of carragheen or carragheenin. The carragheen or carragheenin is used as a starch extender, not a substitute, or to permit the use of larger amounts of starch without the sized sheet picking or sticking to the equipment. R.A.

Impregnated Paper
J. T. Popelier *BP* 808,695

Uniform impregnation at both sides of the paper is achieved by passing the web vertically between two rollers, rotating in opposite directions, one of which is mounted on a horizontally slidable bearing and subjected to adjustable hydraulic pressure towards the reel. The liquid is fed into the space between the web and the rolls forming a bath between the rolls and two fixed lateral walls, through which the web passes. Degree of impregnation is controlled by adjusting the liquid level and hence the duration of the impregnating action of the liquid. R.A.

Chemically Treated Paper for Water Writing
E. Messinger *USP* 2,826,511

A sheet of conventional writing paper is treated with at least two chemical layers which on reaction in an aqueous medium give a coloured product. The resulting paper can be used for writing or typing by means of any suitable instrument using water in place of ink. In an example the paper is first treated with a mixture of an aq. iodide (1) and water-soluble starch (7–15). After drying, a second layer of pure gelatine is applied. When this is dry, a third layer of a satd. cupric chloride soln. in 99% isopropanol is applied. R.A.

Patterned Laminated Paper
Riegel Paper Corpn. *USP* 2,830,005

A laminated wrapping paper with a distinctive alligator pattern is produced by the application of radiant heat at 125–250°F. for 0·01–5 sec. to a laminate of a thin poly-ethylene film to a wax coated base paper. The pattern becomes particularly well defined when the base paper has been dry-waxed, that is, impregnated with wax before being surface-coated with wax. R.A.

Blue Print Paper (IX p. 232)

Wrinkle Coating or Film-forming Composition (XIII p. 236)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Relationship between Structure and Technical Characteristics of Anionic Dyes
I. Mustea *Ind. Uşoară* (Bucharest), **5**, 207–210 (1958): *Chem. Abs.*, **53**, 720 (10 Jan. 1959)

The theory of Otto (*Chem. Abs.*, **48**, 9194 (1949)) that penetration of dyes into leather is increased by increase of conjugated double bonds is confirmed but it is pointed out that the variability of the concentration of pure dye in commercial samples often overshadows structural differences between the dyes. C.O.C.

Mechanical Properties of Casein Films
V. I. Eliseeva and E. V. Kuz'mina
Zhur. priklad. khim., **31**, 1245–1251 (Aug. 1958)

The introduction of sulphonated castor-oil type detergent into a casein film produced by drying its ammoniacal soln. does not bring about any appreciable change in the degree of asymmetry of its molecules. It follows therefore that the mechanical properties of casein films so treated are altered only very little. Plasticisers which do not dissolve the polymer have a similar effect. The increase in elasticity is due to its moisture content, which on elimination leaves the film brittle. At a constant moisture content glycerol increases elasticity. The introduction of a detergent into a film containing glycerol increases its elasticity. The films become brittle and contract on storage owing to migration of the glycerol. The elasticity of a casein film is directly, and its stability to water inversely, proportional to its moisture content. T.Z.W.

Fur Dyeing
T. Kniesche
Z. ges. Textilind., **60**, (19), 872–874 (Oct. 1958)

A description of the structure of furs and their preliminary treatment is followed by a discussion of the various dyeing processes. J.C.F.

PATENTS

Malonaldehyde and Succinaldehyde as Tanning Agents
U.S. Secretary of the Army *USP* 2,851,329

Tanning with an aqueous solution of a saturated aliphatic dialdehyde of > 2 C yields a plump and pliable leather, resembling vegetable-tanned leather, providing the pH is kept between 2·5 and 5·0. C.O.C.

Flexible Leather for Clothing or Shoe Uppers
J. Plot *BP* 808,257

The permeability of leather to gases and its flexibility are improved if the reverse face of the leather is provided with numerous uniformly distributed fine holes extending into but not through the leather. A tool for forming these holes is described. The process is particularly applicable to suède leather. C.O.C.

XIII—RUBBER; RESINS; PLASTICS

Polymerisation of Vinylacetylene

I. M. Dolgopolskiĭ, Z. F. Doller, A. P. Yashine, and P. N. Trofimova

Zhur. priklad. khim., **31**, 1234–1240 (Aug. 1958)

The rate of polymerisation of vinylacetylene is a linear function of the temp. and depends upon the kind of initiator used. If the rate at 60°c. is taken as 2, then in the presence of 1% of 2,2′-azodi-isobutyronitrile, at 60°c. it is found to be 4·1, at 70°c. 4·4, and with 2% benzoyl peroxide at 70°c. it is 26. The presence of oxygen doubles the speed of the reaction. The vinylacetylene polymers produced in the presence of initiators are insoluble in benzene. It is impossible to retard the polymerisation by means of inhibitors such as aromatic amines or phenols. These inhibitors do not reverse the reaction. The mol.wt. of the polymers varies between trimer and hexamer. T.Z.W.

PATENTS

Reducing the Tendency to Scorch during Vulcanising

ICI *BP* 808,431

Rubber compositions, particularly those containing Furnace Black (C.I. Pigment Black 7), have reduced tendency to scorch when they contain an accelerator and a compound of formula $R^1[R^2NSO_2X]_n$ ($n = 1$ or 2; R^1 = hydrocarbon or may be H when $n = 1$; R^2 = H or hydrocarbon; when $n = 1$ the $R^1 + R^2$ together with the adjacent N atom may form part of a ring; X = OM (M = metal or NH_4), OR^1 or NR^3R^4 (R^3 and R^4 = H or hydrocarbon and together with the N may form a ring), e.g. sulphamic acid, 4,4′-sulphonyldimorpholine and 1,4-bis-(dimethylsulphamyl)piperazine. These compounds have the further advantage that their use results in non-porous vulcanisates. C.O.C.

Nylon–Carbon Black Moulding Compositions

Polymer Corpn. *USP* 2,849,414–5

Nylon powder of particle size $< 40\ \mu$. is readily miscible with Carbon Black (C.I. Pigment Black 6 and 7) or molybdenum disulphide and can then be moulded sufficiently compactly so that it can be handled. It is then heated to sinter the particles of nylon together without melting them. C.O.C.

Coating for Cured Polyester Surfaces

Tru-Scale *USP* 2,848,350

The surfaces are sprayed with a solution of a polyelectrolyte resin and a plasticiser. This yields a tough adherent coating without need for baking or curing. The polyelectrolyte, e.g. PWM/MA (General Aniline) or Lustrex 820 (Monsanto), and the plasticiser, e.g. Pluronic (Wyandotte Chemical Corp.) are dissolved up separately in the same or different solvents and the two solutions mixed, the solvent being preferably a ketone, e.g. methyl ethylketone. C.O.C.

3,6-Di-n-propylcatechol as Gelation Inhibitor

Pittsburgh Plate Glass Co. *BP* 807,636

3,6-Di-n-propylcatechol is a gelation inhibitor for precondensates of monomers containing $> C{:}CH_2$ groups and polyesters of $\alpha\beta$-ethylenic dicarboxylic acids and dihydric alcohols. Unlike hydroquinone it neither tends to cause discoloration, only slightly reduces the rate of ultimate cure and its inhibiting characteristics are not affected by pigments such as Carbon Black. C.O.C.

Polyurethanes for Coating Cloth and for producing Fibres and Films

Union Carbide Corpn. *BP* 807,808

Products which form flexible films are obtained by reacting a mixture of primary and secondary aliphatic glycols each having > 3 C atoms between the OH groups, with an aliphatic di-isocyanate, aliphatic di-isothiocyanate or a mixed aliphatic isocyanate aliphatic isothiocyanate containing 2 N:C:X groups (X = O or S) separated by > 3 C atoms. They can be converted into fibres or films or used as coating agents for cloth, paper, leather or metal. C.O.C.

Wrinkle Coating or Film-forming Composition

B. F. Goodrich Co. *USP* 2,849,332

Wrinkle finishes may be obtained directly by fusing a plasticised halogenoethylene polymer formulation containing a small amount of a compatible reactive polyolefinic material, e.g. the compound formed by treating glycerol with pentenoic acid $H_2C{:}CH{\cdot}C_2H_4{\cdot}COOH$. C.O.C.

Colour-stabilisation of Acrylonitrile Polymers (VI p. 230)

Diamino-1,2,4-triazoles as Components of Cold-drawn Fibres together with Acrylonitrile and Acrylic or Methacrylic Amides (VI p. 230)

Copolymers of Dialkylaryl Amides of β-Cyanoacrylic Acid or β-Cyanomethacrylic Acid with Acrylonitrile or other Ethylenic Compound (VI p. 230)

Copolymers of Acrylonitrile and the Monoamide of Guanazole and β-Cyanoacrylic Acid (VI p. 230)

XIV—ANALYSIS; TESTING; APPARATUS

Detection of Added Dye in Cold-pressed Orange Oil

J. W. Kesterson, R. Hendrickson, and G. J. Edwards

Am. Perfumer Aromat., **72**, (2), 29–31 (1958): *Chem. Abs.*, **53**, 601 (10 Jan. 1959)

Ultraviolet and infrared absorption analyses are described. C.O.C.

Comparison of Methods for Determining Intrinsic Viscosities from Non-Newtonian Viscosities

K. H. Cram and J. C. Whitwell

Text. Research J., **28**, 849–860 (Oct. 1958)

Using both rotational and capillary viscometers, absolute flow curves were determined for solutions of 0·1–0·3 g. cotton cellulose per 100 ml. in 0·5 M. cuprietylenediamine over a shear stress range of 0·01–1000 dynes/sq.cm. All solutions behaved as Newtonian fluids below a shear stress of 1·0 dyne/sq.cm. Various methods of calculating intrinsic viscosities were examined, but values based on zero shear rate viscosities were required to avoid dependence on viscometric procedure. Intrinsic viscosities based on non-Newtonian viscosities were less satisfactory. S.B.D.

Test Dyeings in the Qualitative Analysis of Fibres

M. Stratmann

Z. ges. Textilind., **60**, (19), 839–841 (Oct. 1958)

Test dyeings with Neocarmin W (Fesago), Neocarmin MS (Fesago), and Karminazurol (Merck) are used to classify fibres according to type, and also to differentiate branded products of the same fibre type. A chart of the colours given by 34 fibres with the three dyes is given. J.C.F.

Cotton Damage—Microscopical Evidence

P.-A. Koch *Z. ges. Textilind.*, **60**, (19), 833–838 (Oct. 1958)

Short lengths of undamaged ripe cotton fibres, mounted in 15% NaOH soln., rapidly develop mushroomlike protuberances at the cut ends of the fibres. The reaction is not affected by ordinary processing conditions, or by mechanical damage, but the effect is progressively reduced as the degree of chemical damage increases and the D.P. of the cellulose falls. Examples of the application of the method to actual cases of damage are given. 19 photomicrographs. J.C.F.

Determination of Carboxyl Groups in Cellulose

J. P. Fleury and E. Schneider

Bull. Inst. Text. France, (75), 43–54 (June 1958)

0·2–1 g. of cellulose, depending on the carboxyl content, is packed into a column and demineralised by passing 50 ml. of 0·1 N-HCl through the column. After washing with 50 ml. of water, 50 ml. of 0·1 N-NaCl–0·005 N. sodium acetate is passed through the column, and the effluent solution collected. The column is washed with water until the total volume of effluent is 100 ml. The base taken up by the cellulose is determined by titrating aliquots of the initial and effluent solutions with 0·01 N-HCl to Methyl Red. J.C.F.

Determination of Carboxyl Groups in Cellulosic Fibres

S. Schwenkedel *Textil Praxis*, **13**, 1268–1272 (Dec. 1958)

The number of carboxyl groups in cotton and viscose rayon cellulose can be estimated accurately by complexometric titrations with Ca^{2+}, Mg^{2+}, and Zn^{2+} ions as well as potentiometric titrations using Ag^+ and Tl^+ acetate soln. L.A.T.

Fat Content of Textiles (VI p. 229)

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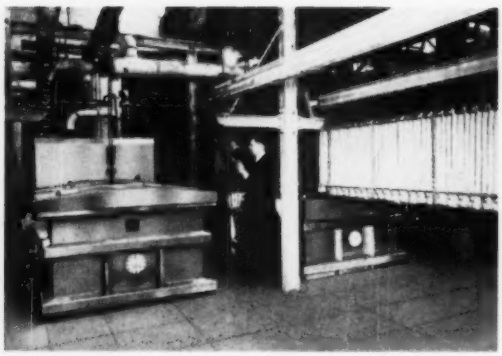
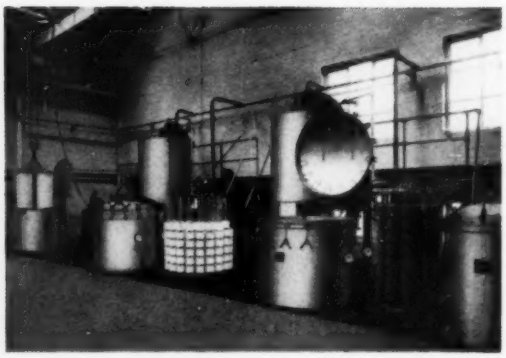


Hank Dyeing machines incorporating the Brown/Longclose double stick system (patent)



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All inquiries relating to Advertisements should be addressed to THE GENERAL SECRETARY, THE SOCIETY of DYERS and COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE.

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ERRATUM

Members' Changes of Address. March 1959 issue of the Society's Journal. Page xxxviii.

Bienkiewicz, J., *formerly* Biedawski, J., of 61 Somersby Road, Mapperley, Nottingham, *to* 19 Cowell Street, Gladesville, N.S.W., Australia

should have read

Bienkiewicz, J., *formerly of* 63 Lees Hill, Sneiton, Nottingham, *to* 19 Cowell Street, Gladesville, N.S.W., Australia

ADDRESSES WANTED FOR RE-DIRECTION OF JOURNALS

The Journals of the Society having been returned through the post it is desired to obtain information for these to be forwarded and any assistance in this respect will be appreciated

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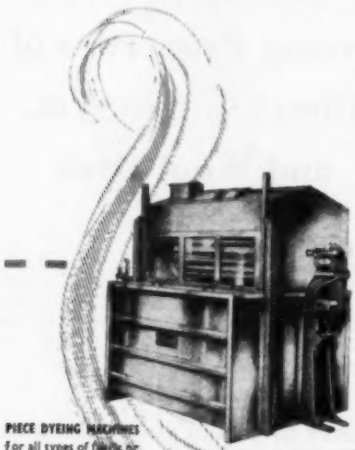

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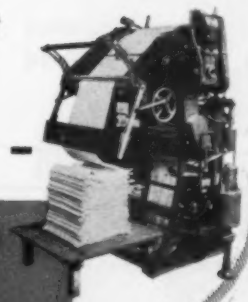

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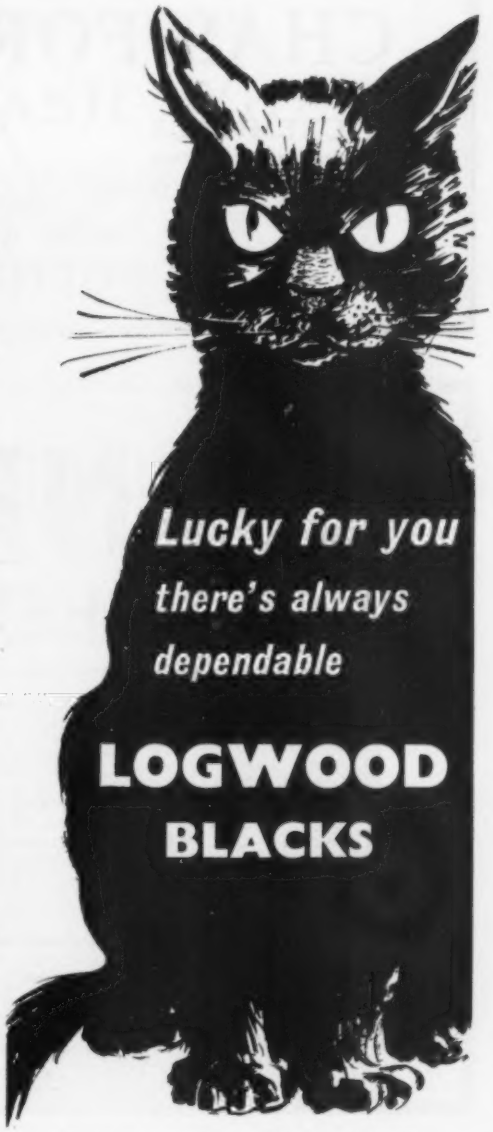

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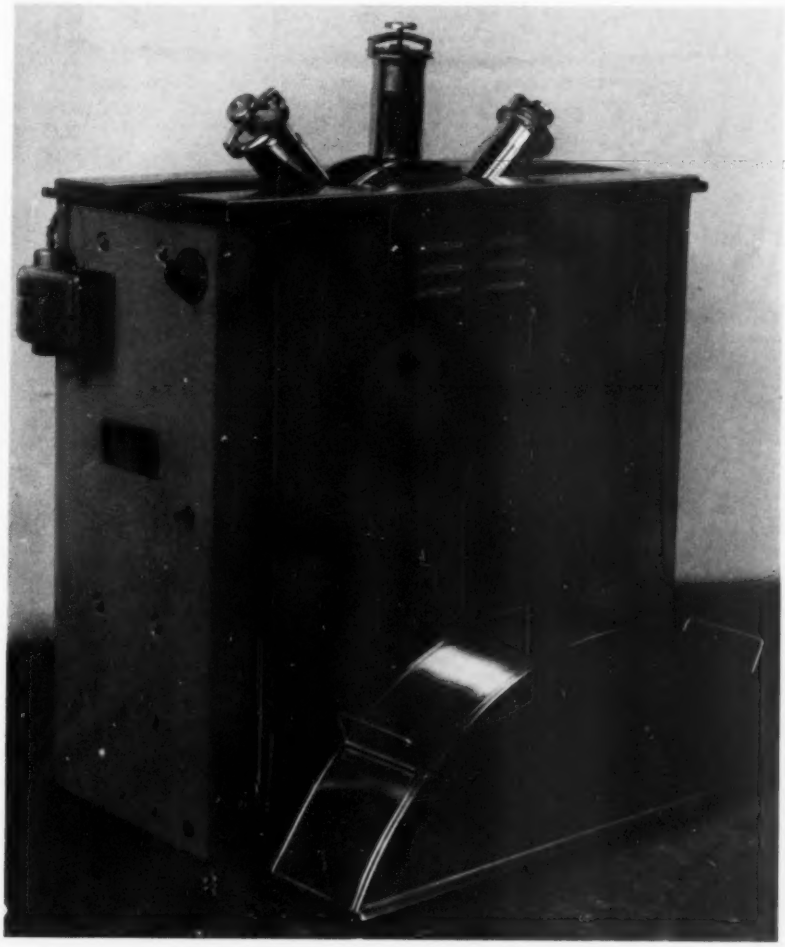

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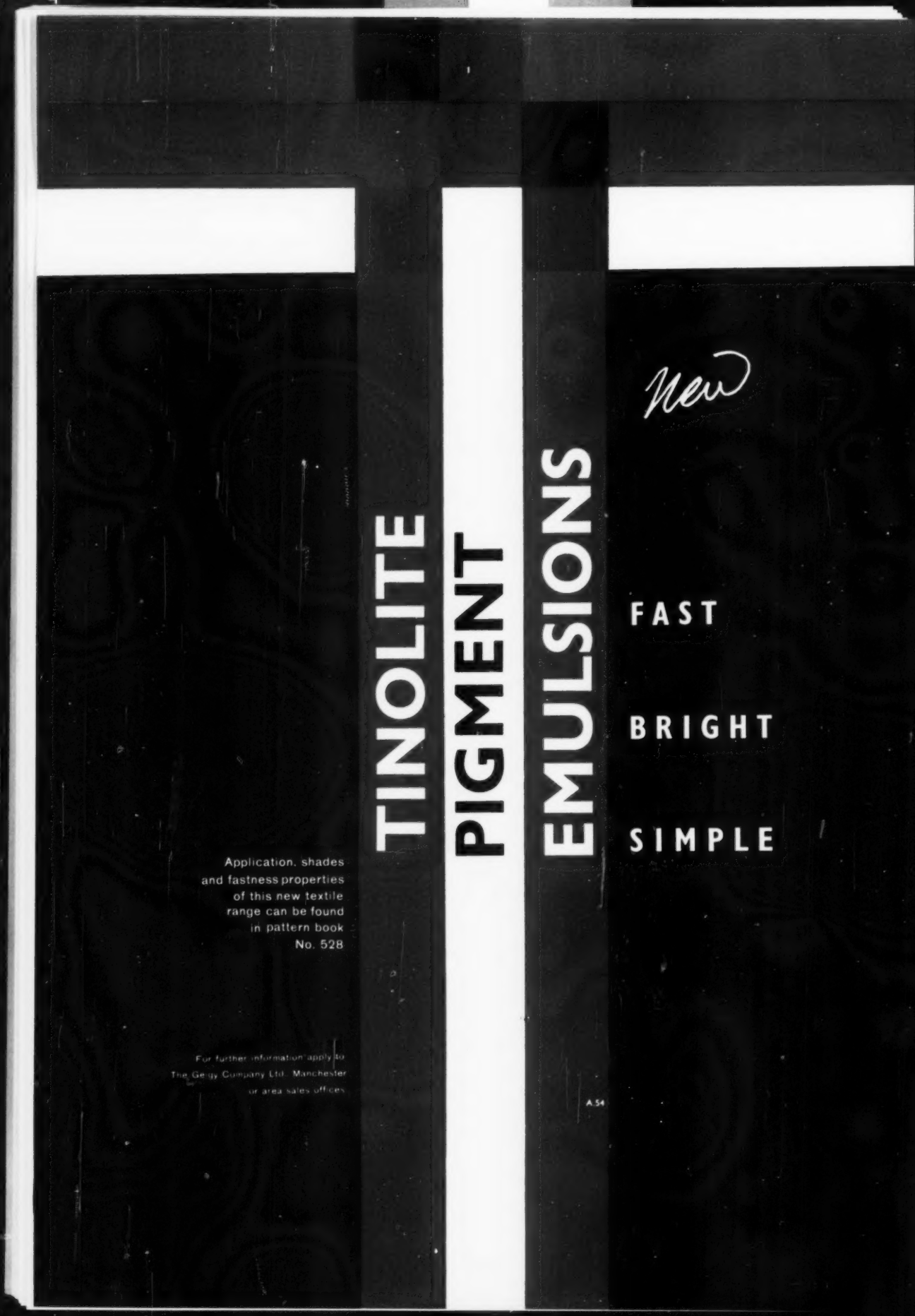
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